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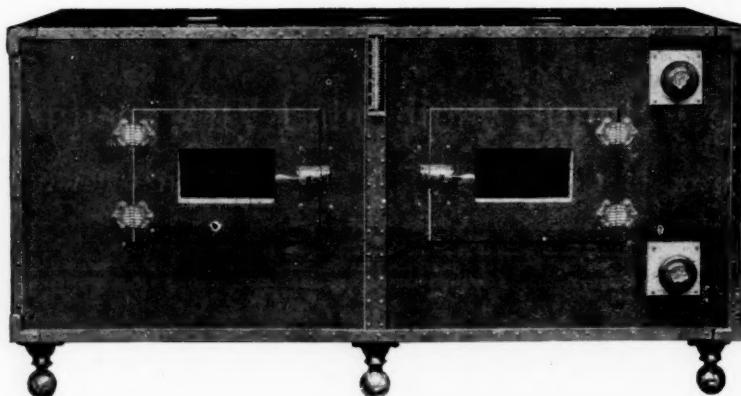
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**VITAMINE REVIEW**

Walter S. Long, City Chemist.

As a preface to this paper, a few explanatory remarks may be in order. These are suggested by an incident which occurred Wednesday. A visitor, apparently much interested in vitamines and also well informed on the subject, requested to see the equipment used by me in my experimental work. I was compelled to make an apology that I make now that my interest in the subject was purely that of a layman, not that of an expert, nor even that of an investigator. However, if there are those here that are as little informed on the subject as I, this review of the work of others may prove of some value, at least to the extent of suggesting matter for discussion.

The subject matter of the paper is in no sense original with me. Certain facts as stated in the literature have been brought together and are presented for your consideration. The writer is not prepared to answer any searching questions.—*The Author.*

Fifteen years ago, Russell H. Chittendon, in his book, "The Nutrition of Man," made the following observations:

"The food of mankind is essentially organic in nature, and while it may be exceedingly varied in character, the principles contained therein are few in number."

"The organic food-stuffs are of three distinct types and are classified under three heads, viz., proteids, carbohydrates and fats. All animal and vegetable foods, whatever their nature and whatever their origin, are composed simply of representatives of one or more of these three classes of food principles."

"There are many suggestions of improvement in bodily health, of greater efficiency in working power, and of greater freedom from disease in a system of dietetics which aims to meet the physiological needs of the body without undue waste of energy and unnecessary drain upon the function of digestion, absorption, excretion and metabolism."

The author in substance states further that advocates of a high "proteid" diet claim that nutrition of the body will be impaired and diseases of various sorts are liable to follow in cases of a lowered "proteid" intake. They cite as an illustration the experience of Oshima with the Japanese navy. The latter had succeeded in stamping out the disease beri beri among Japanese marines by a gradual substitution of bread and meat for the customary rice diet.

"The ordinary food of the Japanese was characterized by great lack of variety, and with such a preponderance of carbohy-

drate materials of a limited kind that it is easy to conceive of a possible dearth of some essential or accessory element necessary for the preservation of that nutritive balance which aids in the protection against disease."

The above views of nutrition may be taken as representative of the general attitude towards the subject during the period just previous to the enunciation of the vitamine theory by Casimer Funk. Only eleven years ago the latter coined a scientific term which today is in common use; a name scarcely old enough to have acquired uniformity of pronunciation, descriptive of that which has commanded the attention of scores of investigators and has been the subject of hundreds of publications.

The scope of the work involved, the widespread interest among men of science and the great volume of literature available are indicated in four books recently published:

"The Vitamine Manual," by Walter H. Eddy, Columbia University.

"The Vitamina," by H. C. Sherman, Columbia University, and S. L. Smith.

"The Vitamines," by Casimer Funk, College of Physicians and Surgeons, New York City, N. Y.

"Eddy's Vitamine Manual" is a book of 121 pages, twenty-eight of which, or twenty-three per cent of the whole book, are devoted to bibliography. Analysis of the bibliography show reference to 577 published articles representing two hundred and four authors, not counting numerous co-authors.

The "Vitamines," by Funk, is a book of 475 pages. One hundred and eight pages or 22.7 per cent of the book include the bibliography. This book treats the subject of vitamines in a comprehensive manner and in great detail.

Since 1914 publications treating the subject of vitamines in a general way, number seventy-five. Articles more limited in the scope of their subject matter number over sixteen hundred. The earliest reference goes back to 1906. The latest records were published during the last month.

The index number of chemical abstracts issued in December, 1921, has listed 180 articles under the headings:

Vitamines, Avitaminosis, Beri beri, Pellagra, Scurvy, Xerophthalmia.

The foregoing facts are mentioned merely to call attention to the vast amount of work that has been done on the subject of vitamines and to the widespread interest as manifested by the number engaged in the work.

Probably the most comprehensive treatise on the subject is that of Funk. The first edition of his work was published in

1913. It was an attempt to classify information relative to vitamines and vitamine deficiency diseases. It contains the facts known at the time, the conceptions of the author, and suggestive questions and ideas for future research. It contains a great deal of fragmentary material; some that is purely hypothetical and lacks many facts necessary for a proper linking of the parts. It is a skeleton structure, but built upon a firm foundation. For seven years it has provided stimulus for the work of others and has witnessed great progress in this line of research. The views expressed there have been proved largely to have been correct. The subject has received universal recognition, and has passed the hypothetical stage and is based on a sure foundation; yet there are great gaps to be bridged. Many of these owe their existence to the disposition of investigators to gain credit to themselves by superficial work. Too few are interested in getting at the basis of phenomena. Experimental work has been practically limited to lower animals. The chemistry, physiology and pharmacology of vitamines deserves the same degree of attention as do pathological conditions associated with deficiency diseases.

Funk's original classification of avitaminoses included beri beri, scurvy and Barlow disease, pellagra, sprue, rickets and some others he considered hypothetical avitaminoses. At present beri while rickets are more or less generally acknowledged as such. Pellagra and sprue have not been definitely established as avitaminoses.

The chemistry of vitamines is little known. The clearing up of the subject from a chemical standpoint will require long and painstaking effort. Some work is being carried on in industrial laboratories, and there may be progress that is not being published.

From the standpoint of political economy the vitamines have become of great importance; people may survive on a practically unchanging diet, a diet found through experience and natural selection to be efficient, yet in times of crisis, such as war, such experience becomes useless and people are compelled to seek some other basis of existence. In such cases a knowledge of nutrition can be applied toward the prevention of disease which might otherwise follow.

The term "Vitamine" was introduced by Funk in 1912. The name has become popular. Other expressions, such as "accessory food factors," "food hormones," "nutramine," "auximone;" water-soluble B. and fat-soluble A., have been suggested and used by other authors, but these terms have not been able to displace the original. Doctor Funk used the term to describe a substance which

he believed curative of the disease, beri beri; since then two other such substances have been discovered, viz: vitamine A. and vitamine C. In connection with the study of rickets there is evidence of still a fourth vitamine, referred to as "Vitamine D."

Vitamines as described by Willaman are a "class of substances whose presence is necessary for normal metabolism, but which do not contribute to the requirements of the organism as regards inorganic constituents, nitrogenous substances and energy producing food constituents."

According to the theory of Doctor Funk, the importance of the vitamines is not limited to animal life alone, but belongs to all living matter. The present view is that the power of synthesizing the vitamines is limited to plants. Apparently, however, not all plants possess this power.

Usually "Vitamine B" occurs to the greatest extent in seeds, accompanying varying amounts of "Vitamine A." Vitamine C is generated in germinating seeds.

Among questions unanswered as to the ability of plants to build up vitamines there may be cited the following:

- (a) Do stored up vitamines stimulate further synthesis of vitamines in the growing plant?
- (b) Do stored up vitamines serve to carry the plant to a stage of growth where certain micro-organisms produce the vitamines in symbiosis with the plant?
- (c) Do certain plant structures, such as the leaves, manufacture the vitamines?

The material best suited to the study of Vitamine B. is the yeast plant. Whether this plant can multiply without vitamine has not been decided. There is some evidence to the effect that a substance closely related to Vitamine B., if not identical with it, promotes the growth of yeast. Vitamine A. and Vitamine C. appear to have no effect upon the growth of yeast. In this connection it may be stated that a method of measuring Vitamine B., devised by R. J. William, is based upon the rate of multiplication of yeast cells. From twenty to several thousand cells were found to develop from a single cell in twenty-four hours, depending on the vitamine concentration. Another method based on the rate of formation of carbon dioxide has been devised by Bachmann.

Some types of bacteria will not grow on pure synthetic media, while others will do so. Addition of vitamine in case of the former promotes growth, while in the latter the presence of vitamines has been demonstrated. The condition of growth of various types of bacteria have been studied. Considering the work, as a whole, it is evident that the growth of some types of bacteria is

stimulated by addition of vitamines, although *bacteria in general seem to exist independently of any vitamines supply.* Bacteria, therefore, may be considered the source of vitamine supply, or of the substances from which they are formed, for the whole organized world.

With regard to the relation between vitamines and higher plant forms some experiments performed by Bottomley in 1912 are suggestive. These experiments were conducted for the purpose of determining the effect of certain types of bacteria on humic acid. An alcoholic or water extract of peat inoculated with the bacteria in question was treated with phosphotungstic acid. The precipitate which formed was decomposed and found to act favorably on plant growth. Such growth was maintained even when the cotyledons, the source of reserve vitamine, were removed from the young plant. These results indicate that plants in their early stage of growth are dependent upon a reserve stock of vitamines stored in the seeds. Later growth depends upon a supply produced by symbiosis with bacteria. The latter produces them synthetically from the organic constituents of the soil.

The subject-matter of modern text books on nutrition is based largely on experiments with dogs and rats. The requirement in the case of rats for complete nutrition is as follows:

- (a) A biologically complete protein.
- (b) A carbohydrate, such as starch, sugar or dextrin.
- (c) A certain amount of fat.
- (d) A salt mixture designed to furnish ions of proper kinds and in proper amounts.
- (e) Vitamines A. and B.

A protein, salt mixture and vitamines suffice for dogs and cats. Whether rats would survive on the latter diet has not been demonstrated.

No experimental animal has been known to survive long on a vitamine free diet. Some animals can live without one or another of the vitamines. In support of the theory that vitamines are supplied the best through the agency of symbiotic bacteria may be cited the unsuccessful attempts to maintain life in certain animals kept under sterile conditions. Such experiments have been conducted with chicks, flies, scorpions, lizards, frogs and toads. To summarize briefly our present knowledge relative to vitamines in the animal kingdom, the following outline is submitted. It shows

the type of animals experimented with and the results found as to vitamine requirements:

<i>Type of Animal.</i>	<i>Vitamine Requirements.</i>
Protazao (one celled)	Vitamine B.
Metazao (polycellular)	Result not conclusion
<i>Insects.</i>	
(a) Flies	Vitamine B.
Fish	No conclusive results
<i>Amphibia,</i>	
Frogs	Vitamine B, Vitamine C, not needed
<i>Birds,</i>	
Chickens	Vitamine B, Vitamine C, not needed
Pigeons	Vitamines A & B, Vitamine C, not needed
<i>Mammals</i>	
(a) Rats	A & B Vitamines
(b) Mice	A & B Vitamines
(c) Prairie Dogs	A & B Vitamines
(d) Guinea Pigs	Vitamine C Results not conclusive as to other requirement
(e) Rabbits. Results in conclusive; animal not so sensitive to lack of vitamines.	
(f) Cats. Vitamine B.—Vitamine A. not required.	
(g) Lions. Vitamine A.	
(h) Dogs. Vitamines A. B. C.	
(i) <i>Domestic Animals:</i>	
(1) Sheep and Goats	—Indefinite results
(2) Reindeer	—Incomplete data
(3) Pigs	—Not clear
(4) Horses	—Incomplete data
(5) Cows and Oxen	—Disease resembles avitaminoses, but data inconclusive
(6) Monkeys	B Vitamine. C—Vitamine
(7) Man	—Positive proof to the effect that man requires at least three Vitamines A. B. C. (Anti-beri beri, anthriachitic and anti-scorbutic).

#### *Chemistry of Vitamines*

*Some differences between vitamines and the ferments.* Ferments are destroyed suddenly between the temperatures 50 deg. and 70 deg. C. while the inactivation of vitamine takes place grad-

ually with rise in temperature. (Complete destruction in general at a higher temperature than in the case of ferments.)

There is some evidence that the vitamines compared with ferments have a relatively simple structure.

Vitamines are quite resistant to chemicals and physical manipulation. Some methods of chemical fractionation result in a partial or complete destruction of vitamine activity.

There appears to be some combination protective against destruction. There are some views to the effect that it is an acid which serves this purpose. Funk believes this to be only partially the case. There is the possibility also that such combination is protective against oxidation.

The difficulties encountered in attempts to isolate vitamine are those which necessarily belong to the separation of a minute quantity of any material from a large mass of some other material. The process is complicated by the fact that the obstructing material is cellular in nature, and further by the fact of the great variety of such material.

The methods used in the study of the nature of vitamines include:

- (a) Fractionation
- (b) Solubility and selective absorption
- (c) Testing activity of combinations
- (d) Synthetic experiment

The first has yielded the most instructive results. It has resulted in the obtaining of a vitamine fraction, simple in comparison with the complexity of the original material. Funk was able to isolate some well defined substances possessing marked curative action, but which upon further treatment lost much or all of their activity. He was able to isolate a crystalline substance of constant melting point and retaining a degree of activity. Whether the active substance was present merely as an impurity, however, is a question that has not been settled. Funk believes that the vitamine would not disappear without leaving a trace. Among the substances usually present with Vitamine B. are products having the pyridine ring. There is a possibility that these pyridine derivations are vitamine decomposition products. If this can be shown to be the case, then it may be possible to demonstrate their chemical constitution and finally to synthesize the vitamines themselves.

When pressed yeast is subjected to boiling with 20% sulphuric acid for twenty-four hours, the acid removed with baryta, and the filtrate evaporated to small bulk, the latter possesses marked

curative action. This stability of the vitamines may be explained on the assumption that the latter is basic in character. On this assumption Funk made the following systematic investigation of rice polishings: 50 kilos of rice polishings were extracted with acidified alcohol and the solution obtained concentrated in vacuum. The fatty residue was melted and extracted with hot water. The water solution, itself very active, was treated with sulphuric acid to a concentration of 5%, and finally by addition of phospho tungstic acid. The resulting precipitate was decomposed with barya and the excess barya removed. The filtrate was curative for beri beri pigeons. The solution was free of phosphorous, protein and carbohydrates. The filtrate evaporated to a syrup in vacuum was taken up with alcohol, precipitated with sublimate; the precipitate re-crystallized from water and the crystals decomposed with hydrogen sulphide. The filtrate was found to be active, indicating no reparation. The filtrate was concentrated in vacuum, the residue dissolved in alcohol and precipitated with platinic chloride. Choline was obtained from the precipitate. The alcoholic solution was still active. The solution was treated with silver nitrate in alkaline solution, the filtrate freed from chlorine with silver sulphate, the silver and sulphuric acid removed, and the resulting solution acidified with nitric acid and then treated with silver nitrate till it gave a brown precipitate with barya. The precipitate was washed free of nitric acid decomposed with hydrogen sulphide, barya removed with sulphuric acid and the filtrate concentrated in vacuum. The resulting product was active. By slow evaporation and addition of alcohol a colorless crystalline substance was obtained weighing 0.4 grams. The needle-like crystal melted sharply at 233 deg. C. Four milligrams of the product was tried as a curative dose. Funk claims there was curative action. He gave to the substance the name of beri beri-vitamine to call attention to the fact that it contained nitrogen and was necessary for life.

A later extraction on a large scale from 380 kilograms of rice polishings gave a yield of 2.5 grams of crude product which on recrystallization amounted to 1.8 grams. Analysis showed the product to conform to the formula  $C_{26} H_{20} N_4 O_9$ . The percentage composition is about that of nicotinic acid  $C_6 H_5 O_2 N$ . The picrate also characterize it as nicotinic acid (m-pyridine carbonic acid).

It is to be noticed that Doctor Funk expresses himself very carefully regarding the curative value of this final product.

Similar experiments on yeast resulted in the isolation of three substances, nicotinic acid, and substance 1 and 11 corresponding in composition to formulae  $C_{23} H_8 O_7 N_4$  and  $C_{29} H_{23} O_9 N_5$ .

Relative to the stability of vitamine B. the following facts may be mentioned:

1. Activity is lost when heated to 120 deg. C.
2. Resistant to acid.
3. Activity not affected by diazotization.
4. Not destroyed by reduction with palladium and hydrogen.
5. Not affected by oxidation with platinum and oxygen.  
(7 to 8 hours).
6. Action of X-rays in small doses without effect.  
In large doses destructive.

The demonstration of vitamine B. consists in feeding to young rats a diet complete in everything except the vitamine. If growth is obtained under these conditions the presence of the vitamine is considered established. Numerous methods of estimating vitamine B. have been prepared and tried out with varying results. The most promising is that of William which consists in noting the rate of multiplication of yeast cells when under the influence of the vitamine.

#### VITAMINE A.—Antrichieic Vitamine.

The chemistry of vitamine A. has advanced but little. Attempts to isolate it have been limited to Cod Liver Oil. A lecithide apparently in a pure state has been isolated from the oil, which appears to represent the total active substance, vitamine A.

The findings have not been corroborated by the feeding of animals on a vitamine A. free diet. The demonstration of this vitamine requires a diet free from the substance in question. Young rats supplied with that diet should show no growth. After ten to fourteen days the substance to be tested is added to the diet. If growth occurs then the presence of the vitamin is demonstrated.

There is some evidence to the effect that the amount of the vitamine in seeds is increased during germination. This raises the question whether higher plants have the power of synthesizing them. The results of some work seem to indicate that vitamine A. may be produced by green leaves only in the presence of sunlight while lower organisms devoid of chlorophyl cannot do so.

Very little is known of the chemistry of vitamine C. Its resistance towards heat, storage, and drying appears to vary with its source. Heating at 110 deg. to 120 deg. C. is more destructive than boiling. Dessication accompanying the heating is partially responsible for this effect. Its activity is more readily impaired in air at room temperature than in an incubator at 37 deg. C. Its de-

structibility appears to depend, to some extent, on the acidity of its source.

Chemical fractionation methods have proved unfruitful.

In his work with lime juice Funk demonstrated the presence of vitamine A. and isolated a terpene  $C_{13} H_{24} O_3$ , a purine base,  $C_6 H_7 O_2 N_2$  and a substance of formula  $C_9 H_{18} O_6 N_2$  and stachydrin  $C_7 H_9 O_2 N$ . He suggests that one of these substances may be cleavage product of vitamine C.

Other investigators have found that vitamine C. may be extracted with alcohol.

There is some evidence that vitamine C. is readily oxidized.

Its presence is demonstrated by its curative effect on animals suffering from scurvy.

The stability of vitamines in general, on heating, does not depend so much on the temperature as upon the duration of heating. One explanation advanced is that the ferments present destroy the vitamines. Rapid heating at higher temperature destroys the ferments. The present more generally accepted view is that oxidation is responsible for their destruction.

From thirty to seventy per cent of the vitamine content of food products is lost by discarding the cooking water. Very little is known relative to the effect of dessication on vitamines. Quick drying at high temperature appears to be less destructive than gradual drying at lower temperature.

As might be expected the vitamine content of canned goods depends largely upon the temperature and duration of heating and the acid content of the raw material. Canned tomatoes possess good anti-carbetic properties, while cabbage and beans lose 65% or more of their activity.

Since the time of Liebig (1854) it has been known that milling decreases the nutritive value of grain. The latter stated, "No single foodstuff loses its value so rapidly as whole grain thru the modern process of milling; the whiter the flour the less nutritive value it possesses." As early as 1872 it was known that pigeons die when fed on fine wheat flour. Dogs may live forty days on wheat flour but continue to live on coarse whole grain bread.

Economic conditions resulting from the war have given rise to a great deal of interest in this matter and have called attention to facts long since established. Efforts to utilize the nutritive value of cereals are being made in practically all countries. In most cases vitamine A. and B. are found in a certain portion of the grain; as a consequence modern refining is likely to result in a more or

less complete removal of these factors. Normally there is not much danger of disease from this cause since people obtain the necessary supply of vitamine from other sources. Bread, however, may constitute a large percentage of the diet of many people, either from choice or necessity. In recent years the knowledge as to the location of vitamines in grain has been modified. It has been shown that vitamine B. is located chiefly in the germ; in rice, wheat and corn; partly also in the pericarp. Some investigations seem to show that wheat germ to the extent of fifteen percent added to zweiback is sufficient to supply the deficiency.

This procedure was adopted by the English army as a protection against beri beri.

five days; sterilized rice in nine to sixteen days. Wheat germs

The heat to which flour is subjected in baking has an altering effect on vitamine B. Pigeons fed on barley which has been heated to 1200 C. for 1½ hours died after thirty days with the symptoms of beri beri. White rice produces this disease in eighteen to twenty-heated to 120 deg. C. lose their vitamine activity but not when heated at 100 deg. C. for two hours. It may be concluded from this that baking does not affect vitamine to a great extent. The use of yeast in dough, of course, would tend to supply and difference in this case.

There is considerable evidence to the effect that seeds develop vitamine C. during germination. Whether this fact may be utilized as an economical measure in connection with milling and baking requires to be shown, while polished rice, on account of its lack of vitamine B. may produce the disease beri beri, the question as to whether it contains vitamine A. has not been answered.

Barley is curative of beri beri. It therefore contains vitamine B. It is deficient in vitamine A. These facts also apply to oats. In wheat, vitamine B. occurs in the germ in relatively large quantities; vitamine A. in small quantities, while the endorperm and bran are deficient.

Corn, generally is poor in vitamine A. although certain strongly yellow colored varieties are rich in this vitamine. It contains vitamine C.

Beri beri, scurvy and rickets are diseases which have been established as due to vitamine deficiency. Certain diseases in children, such as tetany, carbohydiate dystrophy and atrophy, together with pellagra, sprue and hunger edema, in adults are doubtless due to such deficiency. Certain pathological conditions are thought to be in part due to vitamine deficiency. These include

kallak, trench sickness, intestinal statis, sterility, hemeralopia, Graves disease, ophthalmia, tuberculosis, leprosy and pneumonia.

Only two days ago there appeared in a local newspaper a front-page article under the caption,

#### A FOURTH VITAMINE FOUND

**Dr. E. V. Collum Announces a Vital Food Discovery.**

An extract from the article reads as follows:

The new vitamine has been determined upon only in the last seven months,..... and the announcement today was the first public information.

The responsibility of the last statement probably rests upon the reporter, for, on pages 204 and 205 of *The Vitamines* by Dr. Funk, appears this statement:

"Recently. Funk and Dubin have corroborated the statement of Emmett and Lucas that the yeast substance is not identical with vitamine B. Now, if the filtrate from the above is repeatedly shaken with larger amounts of fullers earth, the substance acting on yeast —and which we call vitamine D—is likewise almost completely removed. ..... White pigeons get along very well on Vitamine B. alone. Rats require Vitamine D., besides Vitamine B."

—*Kansas City, Mo.*

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### **COMPARATIVE MILLING AND BAKING VALUES OF KANRED, BLACK HULL AND TURKEY RED VARIETIES—OKLAHOMA WHEAT.**

H. F. VAUPEL.

#### **PURPOSE OF INVESTIGATIONS.**

On account of large amount of publicity given Kanred and Black Hull varieties by Farm Publications as to their apparent superiority in yield per acre and test weight per bushel over Turkey Red, we, through previous unfavorable tests of these varieties, deemed it very important to make a more thorough and extensive investigation of these varieties as compared to Turkey Red for milling and baking purposes. The Oklahoma Agricultural and Mechanical College also being desirous of getting some information as to milling and baking values of these varieties to be used at their

Farm Congress sent their Agronomist, A. Deane, to co-operate with us in the following investigations:

County Agents representative of the Hard Winter sections of the state sent in the samples which are found in the following table. It was intended to get this information before the Farm Congress in session at Stillwater the latter part of August to further the interests of the wheat growers in Oklahoma relative to the proper seed wheat. Owing to lack of time not as many samples of each varieties were tested as we at first hoped to, but with samples at hand we feel a fair comparative baking value of the varieties was obtained.

In order to make our data more complete, test weights and commercial grades were determined by Lee Anthis, Federal Grain Inspector.

#### MILLING EXPERIMENTS.

Samples containing 1,500 grams of clean wheat were tempered for 24 hours, the amount of water added being determined by natural moisture content of wheat. The samples were then ground into flour on a small experimental mill and percentage of flour determined. The bran, shorts and flour are separated by bolting. Only one grade, the so-called straight flour was made. Owing to low relative humidity and the extremely hot and dry weather, the evaporation of samples was so great that the yield was determined by taking the amount of flour and feed, rather than the weight of wheat milled. Samples were weighed as soon as possible after being milled. The milling yields and other milling data are shown in Table I.

#### ANALYSIS AND BAKING TESTS

As soon as the milling tests were completed, the flour was put in air tight containers and moisture, ash and protein determinations made. Ash and protein determinations were computed to a standard basis of 13 percent moisture in the flour, as all samples varied noticeably in moisture content. These results are shown in Table II.

TABLE NO. 1. MILLING DATA ON VARIETIES OF WHEAT—OKLAHOMA WHEAT

VARIETY	Lab. No.	Source of Origin Sample	County Agent	Test Wgt. Per Bushel	Commercial Grade	Flour Yield in bu. and lbs. per barrel	
						Moisture After Tempering	Moisture Wheat as Received
Kanred	S-295	Blaine Co.	J. W. Rapp	58. 2 Hd Winter	11.03	12.0	14.6
Jack Hull	S-294	Blaine Co.	J. W. Rapp	62.8 1 Dk Hd Winter	10.82	11.5	14.8
Blauck Hull	S-297	Garfield Co.	S. E. Laird	61.3 1 Dk Hd Winter	10.16	11.0	15.2
Kanred	S-298	Garfield Co.	S. E. Laird	56.2 3 Hd Winter	11.93	12.2	14.7
Turkey Red	S-299	Garfield Co.	S. E. Laird	59.0 2 Hd Winter	10.71	*12.6	14.9
No Variety Noted (Kanred) *3	S-300	Woodward Co.	Robt. Spencer	59.5 2 Hd Winter	10.31	11.0	14.6
Kanred *4	S-301	Canadian Co.	F. K. West	60.1 1 Hd Winter	10.56	10.0	15.0
Black Hull	S-302	Canadian Co.	F. K. West	59. 2 Hd Winter	11.04	10.8	14.0
Turkey Red	S-308	Woodward Co. (Supply)	Sample of car Wheat A. J. Jordon	57.6 3 Hd Winter	12.38	11.0	14.6
Amber Durum *5	S-304	Kiowa	55.6 4 Amber Durum	12.89	11.2	.....	.....
Kanred	S-305	Kiowa	A. J. Jordon	60.5 1 Dk Hd Winter	12.78	11.0	14.7
Exhibit No. 1 (No variety noted)	S-306	Kingfisher Co.	Clyde McFeaters	58. 2 Hd Winter	11.49	10.4	14.8
Exhibit No. 2 (No variety noted)	S-307	Kingfisher Co.	Clyde McFeaters	58. 2 Hd Winter	10.88	11.0	14.8
Turkey Red	S-303	Our Standard Mill Mix		59.8 2 Hd Winter	11.56	11.5	14.2

\*1 Protein equals nitrogen X 5.7—Computed to a basis of 13 percent moisture in wheat.

\*2 Protein on this sample is below standard for milling wheat.

\*3 No variety noted but resembled Kanred samples.

\*4 Sample contained a great deal of yellow berry, which is the millers' chief objection to Kanred wheat.

\*5 Sample sent in for Black Hull. This variety is a Macaroni Wheat and was not milled.

TABLE II. SUMMARY ANALYSIS AND BAKING TEST OF FLOUR FROM THREE VARIETIES, WHEAT  
OKLAHOMA WHEAT

VARIETY	Lab. No.	Source or Origin of Sample	Texture Loaf		Color of Crumb		Quality Dough	
			Weight of Loaf, Grams	Volume of Loaf, Cubic Centimeters	Proving Period, Minutes	Time of Fermenting Period	23390	510 Excel.
Kanred .....	S-295	Blaine Co.	10.95	0.444	9.58	62.5	2 hr. 30 m	48
Black Hull .....	S-296	Blaine Co.	10.69	0.437	9.85	59.	2 hr. 20 m.	46
Black Hull .....	S-297	Garfield Co.	12.32	0.431	8.93	57.	2 hr. 20 m.	46.
Kanred .....	S-298	Garfield Co.	11.00	0.454	10.44	65.	2 hr. 25 m	43
Turkey Red .....	S-299	Garfield Co.	12.59	0.447	9.27	61.	2 hr. 20 m.	50
(No Variety noted) (Kanred) * 2 .....	S-300	Woodward Co.	10.70	0.438	8.84	64.	2 hr. 28 m.	48
Kanred .....	S-301	Canadian Co.	11.69	0.422	9.14	60.5	2 hr. 20 m.	50
Black Hull .....	S-302	Canadian Co.	13.13	0.465	9.88	59.	2 hr. 15 m.	46
Turkey Red .....	S-302	Woodward Co. Supply	11.79	0.449	11.24	65.	2 hr. 25 m.	46
Kanred * 3 .....	S-305	Kiowa Co.	10.84	0.468	11.50	66.5	2 hr. 28 m.	46
Exhibit No. 1 (No Variety noted)	S-306	Kingfisher Co.	11.56	0.432	10.19	62.5	2 hr. 25 m.	47
Exhibit No. 2 (No Variety noted)	S-307	Kingfisher Co.	11.30	0.441	9.29	62.	2 hr. 20 m.	46
Turkey Red * 4 .....	S-308	Standard Mill Mix	10.86	0.42	10.36	64.5	2 hr. 25 m.	46

\*1 Results computed to a basis of 13 percent moisture in the Flour.

\*2 Resembled Kanred and was averaged with Kanred Samples.

\*3 This sample, although prettiest wheat of all samples, baked below expectations, oven rise or "Spring" was poor.

\*4 The Flour of this sample was taken as a check or standard to compare the others by.

## BAKING TESTS

Bread was baked from each sample of wheat milled, using flour of our standard milling wheat (Turkey Red also milled on experimental mill under same conditions) as a check loaf. All comparisons were made with this flour. All varieties were mixed alike using 340 grams (12 oz.) flour and equal quantities of all other ingredients, except water. Temperature of dough out of mixer being 77°—79° F and fermented at 80 degrees F. Each dough was fermented and proved, what in our judgment was the best time for optimum baking quality of respective flour. Dough was proved at 90° F and finally baked at 450°F for 30 minutes. These results are also shown in Table II, Plates I, II and III, reproduced from photographs will help to visualize these results. Plate I and Plate I b (cross section) are two views of the following samples with respective loaf volumes reading from left to right:—

PLATE I.

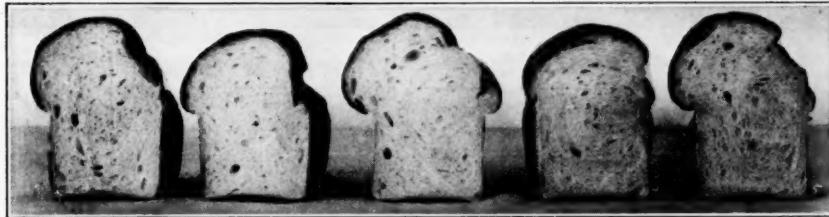
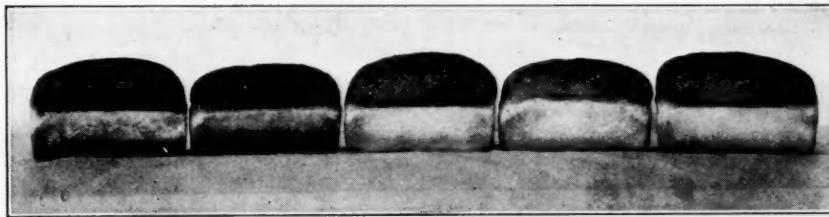


Figure I a and I b.

1. Kanred, Canadian County, 2180cc.
2. Black Hull, Garfield County, 2040 cc.
3. Turkey Red, Check Loaf, 2410cc.
4. No variety noted (Kanred) ? Woodward County 2150 cc.
5. Kanred, Blaine County, 2390 cc.

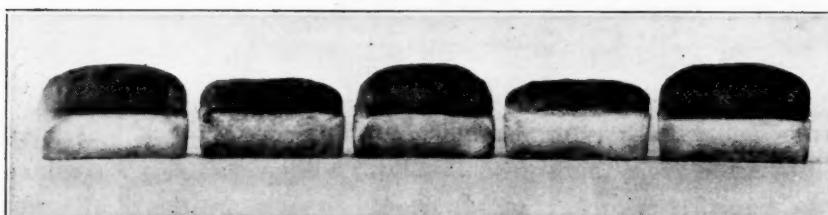


Figure II a and II b.

1. Turkey Red, Garfield County, 2280 cc.
2. Black Hull, Blaine County, 1990 cc.
3. Turkey Red, Check Loaf, 2410 cc.
4. Kanred, Kiowa County, 2140 cc.
5. Turkey Red, Woodward (Supply) 2520 cc.

## PLATE III.

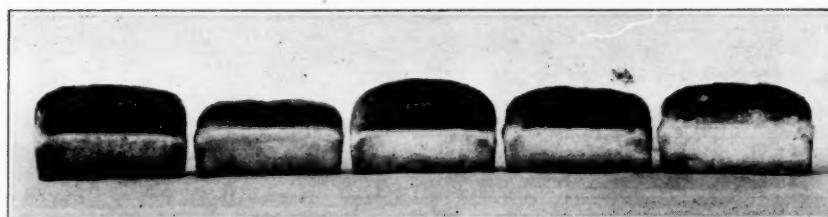


Figure III a and III b.

1. Exhibit No. 1, Kingfisher County, 2380 cc.
2. Black Hull, Canadian County, 1950 cc.
3. Turkey Red, Check Loaf, 2410 cc.
4. Exhibit No. 2, Kingfisher County, 2170 cc.
5. Kanred, Garfield County, 2290 cc.

**SUMMARY OF RESULTS**

The following is a summary of results as to the milling and baking value of the three varieties of hard wheat representative of the hard winter section of the state:—

(1) No variety on the Kingfisher County samples were noted. These samples are not in the averages.

(2) The milling value of Kanred and Turkey were about the same. The one sample of Kanred, which contained a great amount of yellow berry, was rather hard to clean up. This is the millers' chief objection to Kanred wheat, as the majority of samples coming under our observation contained a great amount of yellow berry and consequently run low in protein. The milling yield, as determined on individual samples, may be grossly misleading owing to large source of error in milling small quantities. However, in averaging yields on varieties some information may be obtained. The average milling yield for Kanred and Turkey were about the same, while that of the Black Hull was highest. This is accounted for by the Black Hull having the highest average test weight per bushel. It was noted in milling that the Black Hull was also harder to clean up, resembling the softer wheats in this respect.

(3) The average test weight for Kanred was 58.8 lbs., Black Hull 61.0 lbs. and Turkey Red 58.8 lbs.

(4) The average protein content for Kanred samples was 11.31 percent, Black Hull 10.67 percent and Turkey Red 11.56. Purer strains of Turkey Red averaged about 12 to 12.5 percent, while Black Hull for a dark hard wheat averages between 10.50 to 11. percent.

(5) Average absorption of Kanred samples was 63.1 percent, Black Hull 58.3 percent and Turkey Red 63.5 percent.

(6) Average loaf volume of Kanred samples was 2275 cubic centimeters, Black Hull 1995 cc and Turkey Red 2405 cc.

(7) Flour of Black Hull variety resembles flour of soft wheat varieties when comparing absorption and loaf volumes.

(8) Flour of the Black Hull variety lacks strength—that is, it will not produce a loaf of bread of large volume and of good texture.

(9) Comparing direct baking volumes—Turkey Red proved to be the superior. Some of the individual Kanred samples were equal to the Turkey, but on an average fell noticeably below the average of the Turkey Red. The baking value of the Black Hull as a bread flour proved to be the poorest of the three varieties.

## TEXTURE CLASSIFICATION OF WHEAT

W. L. Frank, Grain and Cotton Exchange, Sherman, Texas, 5-24-22

Several attempts have been made to classify texture in wheat. The most comprehensive classification that has come to the attention of the writer, is that described by Bailey (1). Two factors were considered, namely "pigmentation" and "physical condition or density". Each of these factors was further qualified by descriptive terms. Pigmentation was divided into three classes, namely, (a) Red, (b) Light Red, (c) White. Physical condition or density was divided into four classes, namely, (1) Corneous, (2) subcorneous, (3) substarchy, and (4) starchy. Various combinations of pigmentation and density are possible. In a previous publication Bailey (2) listed the following terms as being used to distinguish between different degrees of texture, namely,—"mealiness", "flintiness", "vitreous", "horny", "Corneous", "Yellow-berry", and "piebald".

The composition of light and dark kernels, from the same samples, was determined by Snyder, (13) who found the average difference in protein content to be 2.65 per cent in favor of the dark colored seeds. This is perhaps the simplest texture classification. In a later publication, (14) Snyder referred to glutenous and starchy kernels, and found the protein content to range from 1.6 to 4.87 per cent in favor of the glutenous kernels.

Freeman (4) refers to "yellow-berry" and "true softness", which perhaps may be considered as a texture classification. In another instance he has explained the "hardness of a wheat", and has discoursed at some length the importance of texture as an indication of quality in wheat. The terms he used were,—"A soft opaque texture" and "a horny translucent grain".

Leith (8) has contrasted the protein content of "the hard berry" with "the yellow berry."

Jones (7) et al, mention the "strictly hard wheats", in connection with their milling investigations.

Shaw and Gaumintz (12) made comparisons of test weight and yield of flour on the basis of the hardness of the kernels.

Hackel (5) used the terms "corneous" and "mealy" in describing texture.

Lyon and Keyser (9) used "horny", "medium yellow berry"

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(1.) Note:—Reference is to literature cited. A list of references is given at end of this paper.

and "markedly yellow berry" in their discussion of the cellular structure of wheat of different degrees of texture.

Roberts and Freeman (10) used the words "*flinty grains*" and "yellow berries" in their classification of Kansas wheat. They also recognized degrees and graduations in texture, for they mentioned the "extremely hard and the completely starchy grains."

Headden (6) refers to the work of Kosutany in Hungary, in which that author discussed the "degree of mealiness", and specifies "flinty, mealy and half mealy wheats". The work of Schinder is also referred to in which "mealy and half mealy kernels" are mentioned.

Bolley (3) has enumerated "off colored, 'spotted', 'white-bellied', 'black pointed', 'pie bald', etc., in his description of deteriorated wheats, and which represents—in part at least—a color classification.

Roberts (11) constructed a mechanical device for measuring hardness in wheat in which the "crushing-point" of individual kernels was determined and averaged for a given sample.

Two designations of texture are used in the Official Grain Standards of the United States for Wheat (16). In the case of Hard Red Winter and Hard Red Spring Wheats, the percentage of "Dark, hard and vitreous kernels" is specified for the three sub-classes in each of the classes named. The expression "per centum of hard (not soft and chalky, kernels)" is used in describing the sub-classes of common white wheat.

Schollenberger and Frank (15) made texture separations on Red Rock—a variety of Soft Red Winter wheat grown in Michigan—in which four degrees were recognized. The classification of kernels was arbitrary, and resembled the texture classification suggested by Bailey. The principal difference in these two classifications was in the manner of describing the degrees of texture. Bailey used corneous, sub-corneous, sub-starchy and starchy while Schollenberger and Frank employed the terms Group 1, of starchy endosperm permitted in each group. The approximate Group 2, Group 3, and Group 4, and further specified the amount fractional part of the exterior surface showing endosperm, was the basis for the classification. The percent of dark, hard, and vitreous kernels, and the mean fractional starchy part of the kernels in each group multiplied by the percent of sample weight in the respective groups, were used in an attempt to show a relation of texture to milling and baking value and to water absorption of Soft Red Winter Wheat flour.

Other references could be cited on the subject, Texture (color) Classification of Wheat. However, those given are representative,

and show what a variety of terms are used in referring to texture (color in wheat). The frequent use of descriptive and definitive terms in discussions on texture (color) of wheat, and the lack of uniformity in the selection of terms is evident. The principal objection to present classifications is that they do not admit of mathematical or percentage comparisons and tabulations.

It seemed that a mathematical expression of relative texture would be better than descriptive or comparative terms having no progressively related numerical value. In as much as it was impractical to assign numerical values to classification terms previously described, a new classification of texture was adopted. The object of the classification was to recognize as many progressive degrees of texture as would admit of easy recognition by a careful worker. Six degrees of texture were recognized, and included:—

1. *Dark Hards* Kernel hard and vitreous and dark in color.
2. *Hard:* Kernel hard and vitreous, but of a lighter color than 1.
3. *Spotted:* One starchy spot visible and affecting not more than one-fifth of exterior surface of kernel.
4. *Mottled:* More than one starchy spot, or one starchy spot visible and affecting more than one-fifth but not more than four-fifths of exterior surface of kernel.
5. *Yellow:* More than four-fifths but not all of surface of kernel appearing soft and chalky.
6. *Starchy:* Entire kernel appearing soft and chalky.

In Table No. 1, relative texture has been expressed in two ways:—*texture index*, and *texture number*. Both expressions are based on the texture classification described above.

#### TEXTURE INDEX

The *texture index* refers specifically to the per cent of a sample by weight, falling into each of the six degrees of texture previously described. To determine the texture index, proceed as follows:

- (a) Separate the original sample into six portions according to the texture classification as given, and record the weight of each portion as per cent of the weight of the original sample.
- (b) Beginning with starchy, record as the right hand figure of the index, the figure in the tens position. (Note: If there is less than ten (10) per cent of starchy, put a zero (0) in the index). That is, if fifteen (15) per cent of the sam-

ple was included in starchy, cut off the figure one (1), and record one (1) as the right hand figure in the index. After cutting off the tens figure (1), in fifteen (15), the figure five (5) remains in units place. Add the units figure—in this case five (5)—to the per cent of starchy, and cut off the tens figure and record as the second figure of the texture index. Assuming the sample to contain twenty-nine (29) per cent of yellow, we would then have five (5) plus (+) twenty-nine (29) equals (=) thirty-four (34). Cut off the tens figure—in this case three (3)—and record as the second figure of the index, thus—31. Add the units (4), to the per cent of mottled (3), and we have a sum of only seven (7). This number being less than ten (10), a zero is recorded in the texture index, which is 031. Add the units figure (7), to the per cent of spotted (14) and we have the sum (21). Cut off the tens figure (2), record in dex, which now reads 2031. Adding the units figure (1), to the per cent of Hard (12), gives a sum of (13). Cut off the tens figure (1), and record as the fifth figure in the index, thus—12031; and add the units figure (3), to the per cent of Dark Hard (27), which gives a total of (30). Cut off the tens figure (3), and record as the last figure in the index, thus,—312031.

The sum of all figures in the index is ten (10). The larger the numerical value of the index, the more vitreous the sample. A glance at the texture discloses the relative texture of a sample by showing the approximate percentage of each of six degrees of texture. To illustrate this point, refer to the texture indexes of Samples H and D in Table 1. The former is represented by the number 112,213 and the latter by 621,000. Sample H and a low texture index, was quite soft and starchy, and graded Red Spring on account of 19.76 per cent d. h and v. kernels. Sample D had a high texture index, was hard and vitreous, and graded Dark Northern on account of 75.69 per cent d: h. and v. kernels.

The texture index as described may be of value in the interpretations of scientific investigations where texture is an important consideration. It is not practical in ordinary commercial practice, but may be useful in research work on texture in wheat where milling and baking value are being studied.

#### TEXTURE NUMBER

The texture number is a modification or adaption of the texture index previously described. It is derived by adding the three right hand figures in the texture index, as,—three (3) plus one (1),

TABLE NO. 1—Percentage of texture separates in each sample by weight, texture index, texture number, percent of dark hard and vitreous kernels and the sub-class of each original sample.

SAMPLE	Dark Hard	Hard	Spotted	Mottled	Yellow	Starchy	Texture Index	No.	Percent Dark Hard and Vitreous	Sub-class
A.....	56.27	11.12	11.37	10.68	9.52	1.01	611110	8.2	67.39	Northern
B.....	39.47	8.60	14.39	19.29	14.87	3.38	412210	7.3	48.07	Northern
C.....	22.79	20.92	6.80	9.78	35.21	4.50	321130	6.4	43.71	Northern
D.....	56.79	18.92	10.12	7.49	4.15	2.53	621100	9.1	75.69	Dark Northern
E.....	47.60	16.50	7.80	14.70	9.20	4.20	521110	8.2	64.10	Northern
F.....	2.21	36.85	8.23	23.49	12.75	16.57	131311	5.5	38.96	Northern
G.....	44.61	15.23	13.20	12.37	8.59	6.00	512110	8.2	59.83	Northern
H.....	9.37	10.39	12.53	18.84	17.51	31.36	112213	4.6	19.76	Red
I.....	46.62	22.26	8.96	8.86	7.96	5.34	521110	8.2	68.88	Northern
J.....	20.18	29.27	19.38	16.98	4.50	9 .69	322210	7.3	49.45	Northern
K.....	15.33	28.84	11.43	13.91	8.45	22.04	231112	6.4	44.16	Northern
L.....	39.24	7.57	11.25	25.70	10.46	5.78	411310	6.4	46.81	Northern

plus two (2), equals six (6), in sample H.—112,213. The sum of the three right hand figures is recorded as tenths, thus—.6, or it may be recorded as units, thus —6.0. The sum of the three left hand figures (thousands) is placed at the left of the first sum, as shown herewith,—4.6 or 46, as the case may be. Other variations and combinations are possible, as for instance, expressing the sum of the two left hand figures as tens, and the sum of the four right hand figures as units, thus,—(in sample H) 28.

The texture number is similar to the texture index. It may be of some value in scientific investigations where it is desirable to express relative texture in terms that are simpler than the texture index previously described.

#### *Conclusions*

1. The Texture Index, and Texture Number, may be of use in scientific investigations requiring a combined qualitative and quantitative method or system of expressing relative texture in wheat.
2. The Texture Index and Texture Number provided a mathematical means of expressing qualitatively and quantitatively relative texture in wheat, and which also admits of comparisons by the equivalent of "percentages."
3. Indefinite, abstract, and definitive or descriptive terms are eliminated, making direct comparisons possible between samples, when the texture index is used.

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*Sherman, Texas.*

**"BLEACHING PROCESSES"**

A. R. SASSE

**Nitrogen Peroxide****ALSOP METHOD**

Nitrogen peroxide was probably the first substance used in the bleaching of flour. About 20 years ago the process was introduced and is known as the Alsop Process. The production of the gas depends upon the oxidation of atmospheric nitrogen by the electric arc. Air is brought into the reaction chamber and when it comes in contact with the arc, there is formed nitric oxide which is further oxidized to nitrogen peroxide by the oxygen of the air. The gas is brought in contact with the flour by blowing it into an agitating device, through which the flour passes.

**NORDYKE & MARMON APPLICATION**

Nitrogen peroxide is also prepared by the oxidation of ammonia. The apparatus was manufactured for a short time by Nordyke & Marmon Co. Only a few mills are equipped with the device as manufacture of them was discontinued through the activities of the Alsop Co. The oxidation of the ammonia is affected by the use of a catalyst, which in this case is platinum wire. The apparatus is very simple, consisting principally of a reaction chamber containing the platinum wire. The chamber has an opening which is covered with mica in order to observe the process of the reaction. To start the apparatus it is necessary to heat the platinum to redness and the ammonia and air admitted. The reaction that takes place is so energetic that the platinum is kept at a red heat. It is necessary to have the quantities of ammonia and air properly balanced or the reaction will not be complete. When the proper amount of ammonia and air is used the platinum will remain at a cherry heat. The best proportion of ammonia-air mixture is 9%.

The reaction that takes place is as follows:  $4\text{NH}_3 + 5\text{O}_2 = 4\text{NO} + 6\text{H}_2\text{O}$ . The nitric oxide (NO) that is formed is farther oxidized to nitrogen peroxide when it comes in contact with the oxygen of the air  $4\text{NO} + 4\text{O} = 4\text{NO}_2$ . Water is split off in the reaction and there is an opening in the bottom of the reaction chamber. The gas is brought in contact with the flour in the same way as described in the Alsop Process.

**FOWLER PROCESS**

Another method that is sometimes used in preparing nitrogen peroxide is the chemical method. There is an apparatus on the market known as the Fowler Bleacher. It consists of a wooden

cabinet containing two glass jars fitted with syphons which are used to deliver nitric acid and water in regulated quantities. The nitric acid and water drip upon a concave casting, having a hole in the center so that the products of the reaction may pass thru, and it is caught in a receptacle under the cabinet. The casting is probably made of copper. The reaction that takes place is as follows:  $3 \text{ Cu.} + 8\text{HNO}_3 = 3 \text{ Cu.} (\text{NO}_3)_2 + 2 \text{ NO} + \text{H}_2\text{O}$ . Then  $2 \text{ NO} + \text{atmosphere O}_2 = 2\text{NO}_2$ . A blower is attached to the cabinet and air is blown in to displace the gas and force it in to the flour agitator or conveyor where the flour is bleached. Glass bulbs are used to float upon the nitric acid which are attached to the syphons so as to lower the syphon as the liquid is used. The metal casting must be renewed occasionally as it is dissolved in time, depending on the use it is given.

Having described the three principal methods for the preparation of nitrogen peroxide, I will now describe the application of chlorine.

#### CHLORINE

##### Industrial Appliance Co. Application.

Dr. J. A. Wesner of Chicago was probably the first to make use of chlorine in the bleaching of flour. Nitrosyl chloride was also patented by him in admixture with chlorine. The patents of Dr. Wesner were later taken over by the Industrial Appliance Co., of Chicago and were put into practical use about 10 years ago. The gas offered considerable trouble in finding a suitable control for regulating it accurately, but suitable apparatus was soon developed.

Industrial Appliance Co. make use of diaphragm valves in controlling the gas, which is delivered from steel cylinders. The indicators used are pressure gauges graduated in ounces per barrel of flour. In order to avoid any chance of overbleaching, there is placed in the outflow a small metallic disc which has a fixed orifice so that the maximum amount of pressure will not permit enough gas to pass through to injure the flour. Discs with different size holes are used, depending upon the amount of flour to be treated. The gas supplied is delivered in steel cylinders under pressure and is known as Beta-Chlora. It is said to be a mixture of chlorine and nitrosyl chloride. One half to one ounce of the gas is used to one barrel of flour, depending upon the nature of the flour to be treated.

#### WILLIAMS BLEACHER

The Williams Process depends upon the electrolytic decomposition of common salt. Chlorine is given off at the positive pole and Hydrogen at the negative, while caustic soda remains in the cell. The chlorine is used as generated. The control of the gas de-

pends upon the size of the cells and the amphrage used. By controlling th current with a rheostat the proper amount of gas may be produced.

Chlorine has found many uses in the last few years, such as bleaching in the milling, textile and other industries, and in the sterilization of municipal water supplies. The electrolytic process is in use on a very large scale at Niagara Falls, N. Y., where most of the supply of chlorine is obtained. It is marketed in steel cylinders which contain from 100 to 200 lbs. of the liquified gas. With the use of suitable regulators the chlorine may be applied to the flour in definite quantities.

#### MILLER CHLORINE REGULATOR

The Miller Regulator is found in many mills for the control of chlorine gas. The machine will reduce the pressure of the gas from the sylinder and affects a control by the use of a needle point valve which is operated by a float. The construction is rugged and is made of a metal that will resist the corrosive action of the gas to a marked degree. Upon opening the machine it will be found to consist of two chambers that are connected with each other by an opening in the lower part of the casting. The machine is partly filled with sulphuric acid which is necessary to operate the float. The gas enters the left hand chamber, up into a sort of a tower, which extends about two feet above the machine and which contains the float arrangement. When the pressure of the gas is lowered by the outflow of gas in the left hand chamber, the float is lowered, which opens the needle valve and permits more gas to enter. In this way the pressure in the chamber is kept constant and with the pressure gauge tubes that are used to indicate the amount of gas that is being used, a very uniform bleach may be obtained. These tubes are a modification of a U tube pressure guage.

The number of inches the liquid is spread within the tubes is an index of the quantity of gas passing thru them. The machine is built to carry from one to five gauge tubes, so that as many as five streams of flour may be treated. The same machine may be used for the control of ammonia gas by merely using iron valves instead of brass. Ordinary lubricating oil is used in place of the sulphuric acid when used for ammonia.

#### FEGAN & SASSE PROCESS

The Fegan & Sasse Process consists of treating the flour in an agitator with chlorine and ammonia simultaneously or by adding the chlorine first and then the ammonia. In the first case, the reaction would be  $8 \text{ NH}_3 + 6 \text{ Cl}_2 = 6 \text{ NH}_4\text{Cl} + 2\text{N}_2$ , and in the

second,  $2 \text{ Cl} + \text{H}_2\text{O} = 2 \text{ HCl} + \text{O}$ . then the  $\text{HCl} + \text{NH}_3$   $\text{NH}_4\text{Cl}$ . The gases are controlled by the use of the Miller Regulator. About 6/10 of an ounce of chlorine is used and 1/10 of an ounce of ammonia per bbl. of flour. The quantity of ammonia may be increased up to the theoretical quantity with which it combines with chlorine. There is introduced in the flour a small quantity of ammonium chloride that serves as a yeast food. The flour may be bleached to a higher degree with this process without destroying the gluten.

#### AGENE PROCESS

One of the latest processes that has come into use is that of Dr. J. C. Baker and is known as the Agene Process. I have not seen the process in use but the description given in the patent papers states that chlorine is passed into a solution of an ammonium salt and the resulting nitrogen trichloride that is formed is removed from the solution by passing a current of air thru it. As Dr. Baker will read a paper on this process, no further mention of it need be given here. (See Jour. of Am. Assoc. of Cereal Chemists VII, 3, 108.)

#### APPLICATION OF GASES TO FLOUR

To apply any of the gases in use for bleaching the methods that are used consist in bringing the gas in contact with the flour in an ordinary conveyor or better, an agitator, made especially for the purpose. The advantage of the agitator being that the flour stream is broken up in order to expose more surface to the action of the gas. The result being a more uniformly bleached flour with a saving of gas.

#### NOVADEL PROCESS

All of the bleaching systems so far described make use of gases for the active agent. A new system which is known as the Novadel Process makes use of a solid substance. The product is mixed with flour in the proportion of 1 lb. to 1 barrel of flour and this is the proportion of 1 lb. to 1 barrel of flour and this is fed into the middlings during the milling process. One pound of the preparation will bleach about 35 barrels of flour. On account of the higher temperature in the milling apparatus the bleaching is accelerated. Benzoyl peroxide  $C_{14} H_{10} O_4$  is at present the chief ingredient of the preparation which is sold under the trade name of Novadelox B. When the decomposition of the peroxide takes place, oxygen is liberated and leaves a residue of benzoic acid which is said to sublime and traces left are expelled during the baking of the bread. The benzoic acid is a harmless germicide and it is claimed by the manufacturer that it will destroy the eggs of insects which are com-

monly found in flour. Bacteria are also destroyed. When not applied during the milling but on finished flour it will require about two days for the complete decomposition of the peroxide to take place.

In conclusion I wish to state that I have attempted to give only a brief description of all of the processes that are now in use for the bleaching of flour as it would not be possible to describe them all in detail in the time allowed for this paper.

*Kansas City, Mo.,  
Southwestern Milling Co., Inc.,*

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### THE NOVADEL PROCESS.

CHAS. T. STORK.

Read before the annual meeting of A. A. C. C. June, 1922.

I have been requested to give some more information about the Novadel Process in addition to what is contained in Mr. Sasse's paper.

I am not a chemist and therefore would not be able to properly answer too technical questions. Questions with regard to the actual practical application of the processes or the results obtained with it in commercial operations will be gladly answered.

The purely chemical information has been given to me by our Chemist in Holland. The practical information is based on results obtained in this country.

As stated by Mr. Sasse, The Novadel process uses a solid substance instead of a gas.

This solid substance is highly active organic peroxide (our patents cover all the organic and inorganic peroxides) which decomposes best with a little higher temperature. This is one of the reasons that we apply the material during the milling. Another reason is the more thorough mixing that can be obtained in that way without the use of extra power or special agitators or other equipment.

Novadelox does not affect the chemical composition of the flour in any manner according to determinations made, which includes moisture, ash, protein, water soluble protein, gliadin, acidity and hydrogen concentration. It does affect the colloidal condition of the protein as it increases the elasticity of the gluten resulting in larger loaf volume and better texture.

It does affect the absorption, the flour increasing this about 1%.

Its use is new as a bleaching agent applied to flour (it has been used for years to bleach vegetable and animal oils). Novadelox is

very strong, it is possible to completely remove all color. At the same time no excess of the reagent, however great, can spoil the gluten or cause deterioration of the flour.

Novadelox is successfully used to bleach rye flour which heretofore had been difficult material to bleach.

We find in practice that Novadelox is already pointed out by Mr. Sasse has one extra advantage beside the bleaching and maturing action.

It sterilizes the flour, giving the flour and the bread baked from it better keeping quality.

Benzoyl peroxide and several other peroxides in the same class are strongly germicidal; solutions corresponding to 0.005% of active oxygen destroyed all known disease producing bacteria and even spores of *bacterium mesentericus* (which are not killed by 5% phenol) within a minute, while hydrogen peroxide solution containing ten times as much (0.05%) of active oxygen was without action on many bacteria even in 60 minutes.

The sterilization has no effect on the bacteria which cause fermentation and therefore the use of this peroxide does not affect the yeast action.

On account of the sterilizing action, Novadelox is being used to good advantage in the manufacture of pancake flour and other package flours. No additional sterilization by heat, electricity or chlorine is necessary beside a treatment with Novadelox. It is also of importance for flour exported to tropical climates.

Novadelox is shipped in tin cans, it has not corroding action on metal, cloth or wood, and does not decrease in strength. We have tested Novadelox that has been kept both dry and in water for over one year and found that the strength was just the same after the storage as originally.

The use of Novadelox-B which is pure Novadelox with a by-mixture of calcium bi-phosphate, is positively without any danger in the mill. The material is non-inflammable.

The Novadel process has been in daily use in Holland for over two years, in England and Germany for about six months and in the short time that we have been able to demonstrate in American Mills, we obtained very satisfactory results.

—Noury & Van der Lande,  
Buffalo, N. Y.

**FLOUR BLEACHING.****DISCUSSION**

Question: Having heard this, I think it would be well to hear about the Alsop process. Here is one Alsop man here.

Mr. MARMADUKE: Well, that is hardly a fair proposition. We haven't any of our technical men who are here, just some from the sales end, but our process, I feel, amongst chemists is pretty well known. We have been through the court—if they will take down the English decision, I will give them the right reference to it—We have fought through the court; what happens to the flour, what is claimed for the process and so forth, is set forth in those decisions, I think much better than I could put them forward, or even perhaps our consulting chemist could. So I don't feel any defense or elaboration of our process is necessary. I thank you.

MR. LEWELLIN: I should like to have had one of the representatives of the Alsop process on this program, along with the others, but not knowing or being in touch with their chemists, I didn't write them on this side of the proposition, but it certainly would have been interesting to take up their particular bleaching process along with the rest of them.

QUESTION: Dr. Dunlap says the improvement in the low grades is caused by the increase in the acidity, and Dr. Baker says he also gets an improvement in his low grades, yet no increase at all to speak of in the acidity. Now, I don't see how those two statements can be reconciled.

DR. BAKER: I explained that in my remarks. The difference between the two processes in that respect is due to the fact that the nitrogen trichloride does not increase the solubility of the gluten and does not increase the buffer value of the flour, whereas the application of chlorine does increase the solubility of the nitrogen and also increases the buffer value of the flour. By buffer value is meant the amount of acid required to bring the flour to a certain hydrogen ion, which in baking bread is pH 5. The differences in baking results with the two methods of bleaching negligible. A gene shows somewhat better results on grades below patent, tho there is a very great difference in the amounts of acid added in the two processes there doesn't seem to be much difference in the final results they arrive at.

QUESTION: I would like to ask the Doctor, or some of the gentlemen, a question. What would be the average pH value for natural hard wheat flour of the different grades?

DR. BAKER: I think Dr. Bailey can answer that question better than I can.

DR. BAILEY: State the question again.

QUESTION: What would be the natural pH value for the different grades of flour—hard wheat flour?

DR. BAILEY: The pH value in terms of pH is higher, that is, in low grade flour, or lower, as the case may be, due to higher buffer action of the low grades. The higher grades we have examined have a buffer value, and clear grades also, depending upon the percentage, ranging 6.2 up to 6.5. I don't recall any that run above that in pH. As age increases pH diminishes, especially in patent flours, because of the low buffer value.

QUESTION: Mr. Chairman, I would like to ask if any work has been done by Dr. Bailey in relationship between the hydrogen ion concentration and amount of soluble protein in flour? I think it is a well known fact the higher grades of flour have a higher percentage of water soluble protein than the lower grades. Is there any relationship?

DR. BAILEY: I don't recall having accumulated any data that I can answer that question directly. I should say offhand, however, that the ordinary ranges of hydrogen ion concentration would not be reflected in any material change in the percentage of soluble protein. The reason I arrive at that conclusion is this: While it is true that a slight increase in acidity might increase the solubility of certain protein in the flour, it would simultaneously tend to diminish the solubility of certain other proteins in the flour, and with the two actions working in opposite directions, I doubt if there would be any material difference in that particular, in the range we ordinarily encounter. Of course, you could go so far as to make a very material change in the solubility of the protein. That isn't ordinarily the case.

PRESIDENT LAWELLIN: I would just like to call the chemist's attention to the fact that Dr. Bailey will speak to us Thursday afternoon on hydrogen ions, and I wish you would reserve questions on hydrogen ion until that time and at this time ask questions on bleaching. Try to confine your remarks to bleaching, so that we will keep a continuity of subject and get the value out of Dr. Baker and Dr. Dunlap.

QUESTION: In that case, I would like to ask if there is a noticeable change or diminution in the amount of enzymes in the flour to the extent that the resultant color is low. My understanding is that when you bleach

beyond a certain quantity with chlorine, there is a decrease in the bloom of the resultant bread. I would like to know if they have performed any experiments or done any investigations to prove that. I am satisfied myself.

DR. DUNLAP: I don't know I quite follow the question which you have asked.

QUESTION: You stated in your address here that there was a diminution in the amount of enzymes when flour was treated by chlorine?

DR. DUNLAP: Yes, sir; they are, ranging as is shown by the constant acidity in such treatment by chlorine.

QUESTION: Does chlorine affect the fermentation, or does it affected the zymase or what the enzymase are affected, and in what quantities?

DR. DUNLAP: The acidity of the flour, as I tried to point out in the paper, is due to probably those things, probably most largely to the enzyme of the ptalin or zymase.

Now on storage I think that there is a continuity of action, that is in treated flour, of this zymase, because we do know that the acidity does constantly increase, keeps on increasing if we store for months, and if it is due to that, then you can see the arresting of that action would destroy the increase in acidity that the continuity of acidity is due to the arresting of those enzymes which bring about an increase in acidity. Now just how much is due to other enzymes and how much is due to the ptalin enzyme I don't know, but I think it is generally assumed that most of the increase in acidity in storage is due to the phosphoric acid. I don't know whether that answers your question or not.

QUESTION: That doesn't answer my question exactly. What I am interested in is whether or not the bleaching chlorine arrests the proteolytic enzymes in the flour. That is the question I have in mind.

DR. DUNLAP: I have no doubt—I have not performed any particular experiments on it but it is quite likely their action would be arrested just the same as the action of the zymase, but I can't say positively as I haven't performed and experiments. I am simply reasoning from analogy.

QUESTION: If chlorine does arrest the action of particular enzymes, I think, when treated with chlorine, in that respect it is deleterious.

DR. DUNLAP: The final answer, as I have often had occasion to say, as to what the flour will do is when it is baked under proper conditions, and what your loaf will show. And there is

absolutely no question that the effect of the chlorine and that change in the acidity produces those effects I mentioned in the course of my paper, widening the decrease in the time of fermentation, the increase in volume, and it is accepted practically everywhere that where you increase the volume, you increase all those other characters which go to make up a proper loaf of bread, namely, increase in bloom, texture and character—they all go hand in hand. So that the changes which take place, the arresting of the enzymatic changes which might take place because of material in the flour before treatment, after treatment certainly do not affect the character of the loaf, which, after all is said and done, is the final answer as to what your flour will do, and there is no question about the increase in volume; no question about the increase in the proper bloom, the porosity, the texture and all of flour, chlorine treated. That I think is the only question. Now, does that give you my point of view?

PRESIDENT LAWELLIN: I would like to hear the millers take a little part in this discussion.

QUESTION: I thought I heard some remark about the question of chlorine destroying bacteria and molds in flour. I would like to know if there is any experimental data or practical demonstration upon that?

DR. DUNLAP: There is no data which has been published. I have my own experimental data on that and we have carried on experiments for a period of several years, not only on different grades of flour but on all types of cereal products. Our results have shown beyond question that, where there is no opportunity for infestation from without, that flour which has been treated with chlorine does keep very materially better, and other cereal products as well keep very materially better, than untreated flour. The question of the keeping quality of flour through chlorine treatment first came to our attention a good many years ago when we received word from the South, where storage conditions frequently are rather adverse to the proper keeping quality of flour, that the flour which had been treated with chlorine would keep very much better than untreated flour, much more free from mold growth and infection from insects, and I am speaking from without, not within, because if flour has been treated with chlorine, if there is a chance of infestation from without, it will become infested just as readily as the flour that has not been treated with chlorine. I hope to carry on this spring some co-operative work with the Bureau of Entomology in Washington on the effect of chlorine in preventing destruction of storage cereals after they have been

treated with chlorine. Of course that has no direct bearing on flour *per se*, but it does have a very marked economic value if any result can be obtained, and so far as the results that I personally have obtained over a period of several years, it looks as if we may get some very valuable results. I hope we do. We must from the economic standpoint.

QUESTION: I would like to ask Dr. Baker and Dr. Dunlap regarding the treatment of bran to prevent super-heating, whether Agene or beta chlora will prevent heating of bran, and how it should be treated. Whether it can be treated with the same method you are now treating the flour.

DR. BAKER: I have done no experiments whatever. I would be very glad to experiment on that material.

QUESTION: I understand there are some mills using that process for treating their bran. Like to know about it.

DR. BAKER: We would be glad to co-operate with you on experiments on that subject.

DR. DUNLAP: I am in the same boat with Dr. Baker. I know nothing about the chlorine treatment of bran.

QUESTION: I would like to ask Dr. Baker and Dr. Dunlap about a case that was called to my attention some few months ago. Perhaps this question had better be directed to the Alsop people, but they have no chemist here, so I will ask the other gentlemen. A miller called my attention to a sample of flour which had been left in the electrifiers overnight. When this sample of flour was examined the next morning, it had turned a pinkish brown, while no such phenomena was noticed in the other flours. They tried this experiment by placing bleached flour in the room with the electrifiers, and also in the mill outside. It was noticed in the morning that the sample of flour that was left in the room with the electrifiers had turned a pinkish brown in color and that this color disappeared on baking, but that the sample left in the mill did not show this effect. As I understand it, this proposition was tried on both patents and clears with the same results, and also with bleached and unbleached flours with the same results. Can anyone tell what the answer is?

MR. MARMADUKE: Did they try that experiment again or just that one time? Did they make a series of experiments?

QUESTION: My understanding was they noted the fact; first tried it on patent flour, bleached and unbleached, and then on grade flour, bleached and unbleached.

MR. MARMADUKE: Did they try several times, say in succession?

QUESTION: I believe these matters were in succession when

they tried the different ones.

MR. MARMADUKE: And another thing—did they note whether they had any leakage of gas in the electrifier room?

QUESTION: I explained to them probably there was a leakage of gas and overbleached on that account.

MR. MARMADUKE: If there was no leakage of gas—all there would be in the electrifier room is simply your heat you know, and it would depend upon the size of the electrifier room. Never personally ever heard of anything like that before.

QUESTION: I never, either. What I wanted more than anything else was to know that if leakage of gas did occur could it go to such an extent as to cause this?

MR. MARMADUKE. I don't think so. Another thing, where did they lay those samples in the electrifier room? Was it on the shelf or on the floor?

QUESTION: I wasn't that much acquainted with that experiment.

MR. MARMADUKE: I would like to know their name.

QUESTION: I don't know myself. It was brought to my attention by the Department of Chemistry in the National Miller.

MR. MARMADUKE: When there has been something unusual happen that way, when you trace it down you generally find it is from some outside cause. There is nothing mysterious about the Alsop process. Nothing mysterious about it. Sometimes you trace those things down you find it is some very simple thing clear outside of the process caused something like that to happen. In the early days of the process you would find maybe some fellow who found a little pink or blue, or something; come to find out, it was paint on the outside of the sides. I would personally like to know the name of that mill, if you can find it out for me.

QUESTION: Perhaps if you see Newt Evans, he can give you some data on that. Now, I don't know it was your process.

MR. MARMADUKE: Well, that was a different story, then. You were talking about an electrifier room.

QUESTION: They just said electrifier, in the room with the electrifiers. Now whether your electrifiers or not, I don't know; whether bleaching electrifiers or not I don't know. I assumed that they were.

Another case that was called to my attention, and that came under chlorine. I was sent a sample of flour by a miller. Previous reports on this sample had been to the effect that the flour was musty. I used every effort I knew of to prove that it was musty and failed to do so. The flour in baking, as reported by the miller—I

didn't bake it myself and didn't see it baked—was that the flour gave a loaf of bread which was decidedly sour and had an acid odor. I ran the free hydrochloric acid on this sample of flour and got 1.85 percent free hydrochloric acid, which would give a decidedly sour loaf of bread. Now, this was by a salt bleacher, I presume. I tried to analyze his conditions, and what I would like to know, is there, or could there be, conditions in the application of chlorine whereby free hydrochloric acid might be formed?

DR. DUNLAP: Well, Mr. L\_\_\_\_\_, I tried to point out in the course of my little paper that the acidity which is produced in flour through treatment of chlorine is due to the development of hydrochloric acid.

QUESTION: Yes, but you misunderstand me, Dr. Dunlap. What I mean is, could there be conditions after the application of your chlorine which could cause the chlorine to go to hydrochloric acid instead of bleaching the flour?

DR. DUNLAP: Oh, no, because just as soon as the chlorine hits the flour it is gone. It either combines with the carotin or with the unsaturated oils and fats and so far as I know the only other changes that take place are the conversion of the resistant chlorine into hydrochloric acid in the way I have indicated. Now, of course, we all know this same use of chlorine aids the products of the unsaturated oils and fats and from what we know of this carotin-like body, this carotin, a body of that kind is perfectly stable under normal conditions, and I can't conceive of any untoward condition arising whereby the acidity would be increased through the formation, after chlorine treatment, of hydrochloric acid.

QUESTION: No, I mean during the placing of the process in the agitator; in other words, in the agitator.

DR. DUNLAP: No, the change that takes place, so far as I know, is as I have just indicated to you. Of course the amount of chlorine that would go to form hydrochloric acid would be dependent upon the amount of chlorine that is used in your agitator, which, as I say, is a varying amount depending entirely upon the character of flour that is to be treated. The larger amount that would be used would necessarily mean that a larger proportion of that would go to your carotin and your unsaturated oils and fats, to your wheat oil. So that you will find in some cases as high as fifty, possibly sixty, per cent. in excess — where larger amounts of chlorine are used, you will find from fifty per cent. chlorine has gone into the formation of additional products with the unsaturated bodies, such as carotin and unsaturated

glycerine, but no change beyond that of hydrochloric acid being formed except as I have just indicated.

QUESTION: Could it be possible for the condition of temperature and humidity to be such that the chlorine would have a greater affinity for hydrogen and the formation of hydrochloric acid than the affinity it would have for carotin?

DR. DUNLAP: I should say not. There is a general rule, whether applicable to this case or not I don't know, but I should say it would be,—the only effect that an increase in temperature, such as one would expect in milling operations, would be to increase the rapidity of the action. Not the direction of the action, because your changes in temperature are not very great. I don't know what it would be; probably 10, 15 or 20 degrees.

QUESTION: Not over that.

DR. DUNLAP: Yes. The general rule is the rapidity of the chemical action would double for every increase of ten degrees. Your temperatures at the upper limit are not very much higher than at the lower limit, so that the only change you will get will be in the rapidity of the action rather than a change in the direction of action. I have never run across a case where the acidity could possibly run as high as that, under normal conditions.

QUESTION: Mr. President, I would like to ask Dr. Baker and Dr. Dunlap what the extent of agitation against bleaching was in New York state and just how far that matter has progressed?

DR. BAKER: I had the privilege of attending all the hearings in New York on the subject of bleaching. Commissioner Cope-land was started by some means or other—possibly our friend Braway McCann—to oppose bleaching. He believes "every human being should eat bread made only from whole-wheat." He states, "I am going to convince the people in New York City they should eat wholewheat bread, and before I am through every one of them will be doing it." That is a rather large statement. As an opening gun for his campaign, for some season or other, he states, "I will start out by prohibiting the bleaching of flour so I can keep people from having white bread." That is the basis of his argument. He is going to shut off white bread as far as he is able so he can make people eat whole wheat bread, if he can stuff it down them. That is his philosophy. I believe we can show him his error and that he is going to attack it from a more sane standpoint.

QUESTION: I would like to ask Dr. Baker a question, if I may. You said you could remove all the color that could be extracted by gasoline.

DR. BAKER: Yes.

QUESTION: Then I infer from that, your treatment will bleach bran articles.

DR. BAKER: No, not at all.

QUESTION: In other words, I don't infer from that you can bleach low grade flour?

DR. BAKER: Only oil soluble color. And possibly a little color that reacts to the additional acid. Now whether these are the same color or not is not certain. You are familiar with the ordinary indicator reactions. When you add acid to an indicator it changes color. Whether that is a characteristic of carotin I am not familiar enough with its properties to say. Dr. Dunlap, are you familiar with that, whether carotin has the action of an indicator or not?

DR. DUNLAP: Carotin, by treatment of acid, like all ferments (ferments) would have a tendency to combine with them and when you break the unsaturated condition, you double the \_\_\_\_\_ and you get away from the color condition and go toward the colorless.

DR. BAKER: Well, that is by the oxidizing power.

DR. DUNLAP: Well, chlorine would be the same, probably hydrochloric acid would be the same.

QUESTION: Mr. President, I notice today for the first time Wolf Company are doing some bleaching, putting out a bleaching equipment and I would like to ask Dr. Hess and Mr. Sasse whether they can tell us what this process is.

PRESIDENT LAWELLIN: Mr. Sasse is not here, but maybe these other—

MR. MARMADUKE: What he has reference to is not Wolf. It is made by the Kansas City Corrugating people. I think what you have reference to is the Miller control.

QUESTION: Dr. Dunlap, in the bleaching with chlorine gas, the absorbtion of the unsaturated compounds of chlorine is also assisted with the hydrochloric acid bleaching action is it not?

DR. DUNLAP: That I can't answer just to what the hydrolysis takes place. I have always assumed—yet it is a very difficult matter to determine in an experimental way—in a thing of that kind one must use their chemical reasoning and chemical analogy rather than—well, it is almost impossible to settle in an experimental way all the changes that take place practically instantly, just as soon as your chlorine hits the flour, but we do know that chlorine and water react and comes to a standstill and equilibrium with the formation of the hydrochloric acid and hydrolysis acid, and we don't know, of course flour enzyme is noth-

ing but organic material which would reduce to hydrolysis acid. With the formation of hydrochloric acid, you disturb the equilibrium forming hydrolysis, and so on to the end that your hydrolysis would practically disappear. It may be that part of that hydrolysis would practically disappear. It may be that part of that hydrolysis acid does react with the unsaturated glycerine.

QUESTION: That is what I was driving at, whether the carotin in breaking up, in splitting up of the hydrochloric acid, whether the carotin would become oxidized, or do both.

DR. DUNLAP: It may possibly have an effect on the carotin.

QUESTION: Some of these compounds can absorb both oxygen—

DR. DUNLAP (interrupting): I would be perfectly safe to say isolate the carotin and treat with hydrochloric acid and you will have no color, but to the extent to which that is carried on, I don't know.

DR. BAILEY: There is another group of substances I should like to offer or refer to this afternoon, and that is another group of substances that seem to have a bleaching effect and that is salts. Persulphuric acid and persulphates are not extensively used in this country although I understand they are used in England quite a bit, and they release oxygen. It is incident to their decomposition, which seems to exert a bleaching effect. I have no particular interest in this group of substances. Merely mention them to make the record complete.

QUESTION: I would like to ask Dr. Baker if he would sum up for the benefit of the members and guests, and friends, how far his process for maturing flour is comparable to natural aging? That is to say, for my own information, and for the information of many others, the question is often put us: How far is the artificial treatment of flour by reagents, such as nitrogen trichloride, comparable to natural aging in which no reagents are applied.

DR. BAKER: Well, in general, there is no bleaching process that is or can be the same as natural aging, for natural ageing is the result of enzyme reaction and probably slow atmospheric oxidation. But the results that bleaching produce are in most respects similar when you come to bake the loaf of bread. In the case of Agene process, my opinion is that the results of bleaching are distinctly superior to those that can be obtained by ageing, that is the improvement in color and in characteristics may be ahead. Now in the Agene process, although it does not increase the acidity, it does bring the hydrogen concentration to practically the same point when properly fermented. That is the important thing, and

not the buffer value which develops by natural aging. I say again, I don't believe any bleaching process can particularly be said to be aging, for it isn't; it is bleaching and maturing. It is done instantly, and there is added to the flour a certain amount of reagent, which is not developed in ageing. I can't see how a man can say bleaching i.e. ageing, tho the effects are similar.

QUESTION: Then according to your statement, as I understand it, if I should ask for an aged flour, under no consideration would you attempt to introduce by some casualistic method of argument, if you would, an artificially treated flour as the product for which I asked?

DR. BAKER: No, I would not. If you want aged flour, it would be aged flour.

QUESTION: And naturally aged flour?

DR. BAKER: Certainly; they are different.

QUESTION: Well, I think that is a very frank statement.

DR. BAKER: Well, it is the truth.

QUESTION: May I ask a question, and call attention to figures that were quoted by Dr. Dunlap this afternoon, comparing the acid numbers of natural flour with chlorine treated flour, and where those numbers progress in the case of natural flour and are constant in the case of bleached flour. In connection with those tables, he gave the loaf volumes that were obtained and if I have put it down right. 2060 cc was given as the volume of the fresh natural flour, and after the 259 days the loaf volume was 2390. In the case of treated flour, the fresh was 2340 and after 259 days it was 2350. Now if you would figure those tables, according to my figuring, the change in volume is taking place in the naturally aged flour amounts about 15 per cent; in the case of treated flour, the increase of volume in that same period is less than .5 of a per cent. It would appear to show that the natural aging of flour has, after 259 days, shown a greater loaf volume than in the case of treated flour, and isasmuch as not every miller is able to sell his flour just as soon as it is made, and there probably might be a good deal of it remain in storage or in the mill for the length of time given here, where is the benefit of the treatment?

DR. DUNLAP: I didn't quite follow your figures. You said that the figures I gave, that the storage after 259 days gave an increased volume of 15%?

QUESTION: No. In the volume—

DR. DUNLAP (interrupting): That was on the net volume.

QUESTION: Yes, the net volume. The natural flour was quoted as having a volume fresh of 2060, and after 259 days of 2390, showing a greater increase of volume through natural agents

than appears through the other volume where the figures were given for the treated flour.

DR. DUNLAP: I think you misunderstand what I was driving at. The point I was trying to bring out was this, the flour that has been treated with chlorine was stabilized. One of the reasons of that stabilization is the constancy of its acidity and one of the results of the stabilization of its acidity is the constancy of its baking value, which I think is brought out very clearly from this table. Now beyond that I don't quite follow the point that you wish to make.

MR. LAWELLIN: Dr. Dunlap, I think I get his point of view. I take this, that in bleached flour, or treated with chlorine—I will say treated flour, you get an increase in volume immediately, while with natural aging you get an increase of fifteen per cent. in 259 days. His question then is.—What is the value of maturing your flour if this increase can be obtained by aging in the small mill where they cannot dispose of the product immediately and it may get the natural aging necessary?

DR. DUNLAP: I tried to bring that out in the course of my paper, from the commercial standpoint because of the question of cost, and particularly for this reason: Flour that has the natural age in constantly becoming better until it reaches its peak, and beyond that point your flour begins to go down hill again. There is no constancy of time that any one can give which will tell you when any type of flour is going to reach that peak. You may reach it in three months, you may reach it in four months, you may reach it in nine months. I have given you one example, the one you have just quoted, where the peak in the baking value had not been reached at nine months. I gave you another example where it had been reached in four months. So you can see that the storage of flour in order to develop it to its highest baking value is a very difficult matter for the small mill particularly, where they haven't the resources to follow the aging of their flour, and that is the thing which is going to change, according to the character of the wheat they use, according to the character of the milling, according to the conditions of tempering and a lot of conditions absolutely unknown to us, all blind except we know the general fact flour does mature or age. Flours do age, they do reach their peak, they do decline, and that that period of time is a period which no one can tell us beforehand.

QUESTION: I was just simply calling attention to the tables as given, and that question came into my mind and I just projected it into the meeting. That was it. I noticed that the highest

acid number given for the natural flour was .135 and the other, which was constant, was .151.

DR. DUNLAP: Oh, is your point this: That you have a lower acidity in natural aging flour than you have in the matured flour?

QUESTION: No, the first thought was we had better baking results from the natural aged flour than we had from treated flour of the same age. That was the point that I had—

DR. DUNLAP: (interrupting): Well, the figures show that; that in this particular case that is shown.

PRESIDENT LAWELLIN: It wouldn't hold true in all cases.

DR. DUNLAP: Oh, absolutely not at all. I didn't bring this out to show what good results you may get with chlorine. All I brought it out was to show flour can be stabilized through this treatment, which stabilized condition lasts for a number of months, certainly long enough for all commercial operations, and that natural aging of flour increases through a period of months, in this case it is nine months before it reaches its optimum, and in another case—but I didn't give you other figures (it wasn't necessary) the optimum point was reached in four months. That was all I meant to bring out by that set of figures.

MR. LAWSON: Mr. President, I believe this argument here, viewed from the economical standpoint of the consumer, admitting for the sake of argument at the end of eight months you get flour baking the same as bleached flour, suppose the man operating a thousand barrel plant had to carry his flour for eight months; now what stock of flour has he got to buy? For the sake of argument, say that it is worth \$5.00 a barrel, and he is paying six per cent. for his money. You have got twenty cents a barrel interest on it. You have got your insurance to put on to it. You have got your loss in weight to put onto it; you have all your losses, and your taxes, and I will venture the assertion that 35c a barrel will not cover the cost of carrying that flour eight months.

PRESIDENT LAWELLIN: That is one of the big arguments for matured flour.

MR. LAWSON: It is dollars and cents that count. I would like to refer a matter to Dr. Dunlap that was referred to me about a year ago by a Nebraska mill, that has since gone broke and been re-organized, so I will mention the name. Well, the name is unimportant. They reported to us they were bleaching flour for Southern export, Industrial Appliances apparatus, and that they had received complaints that after several months the flour had deteriorated appreciably, in quality very much, and they start

ed a series of experiments, so they said, to determine whether that was due to storage conditions or what, and they reported storing flour under what they presumed to be ideal conditions, after three months the chlorine bleached flour was very noticeable for the appearance of the crust on the loaf and after six months they reported the quality was appreciably diminished, and after, I believe it was nine months, nine months or a year, the flour was absolutely worthless, and that flour treated by another process hadn't gone through that. Now they wanted to know if we had ever had that experience. We have this equipment and have never had that experience, and I was just wondering if you could explain why they had it.

DR. DUNLAP: Well, I wouldn't attempt to answer or attempt to give an answer, with certain conditions, without knowing thoroughly everything about it.

MR. LAWSON: Well, I had no detailed information about it.

DR. DUNLAP: I am not going to attempt to set up a lot of straw men and attempt to knock them down. If you can give me all the conditions, I want to know, then I can attempt to answer it, but I wouldn't attempt to answer it offhand. Perhaps a satisfactory answer to it is your own experience.

MR. LAWSON: We never had that experience.

DR. DUNLAP: I think that is sufficient answer there must have been a "nigger in the woodpile" somewhere other than the Industrial Appliance Company.

QUESTION: I may not have heard it, but I would like to ask if it is a fact that you use beta chloral, as I understand you do, and your chlorine is distinctive as bearing on the chlorine on the market?

DR. DUNLAP: Yes. Beta chloral is the trade name under which the gas in the liquefied form is sold. It is made by the Electric Bleaching Gas Company at Niagara Falls, and we have to a certain extent supervision of the production of it. The chlorine is especially purified for this work and there is introduced into it at the filling point a sufficient amount of liquefied nitrosyl chloride to make one half of one per cent. of the total one hundred per cent. of the contents of the steel cylinder. So that the beta chloral which is used is not one hundred per cent. pure chlorine. It is 99½ per cent. pure chlorine and ½ per cent. nitrosyl chlorid. Nitrosyl chlorid, of course, is a very powerful agent for diminishing the coloring of natural flour. One objection to the use of nitrosyl chlorid, *per se*, is that we have never been able to find a satisfactory container. Of course, apart from all our theoretical and scientific

discussion here, there is a commercial side which must be considered by those who are dealing with these processes, and one of the things we have found impossible yet of solution is to find a satisfactory commercial type of container for the handling of pure nitrosyl chlorid. But as an agent *per se* for affecting the acidity and also affecting the carotin, personally I think it stands almost is a class by itself, with due respect to Dr. Baker.

QUESTION: Mr. President, Dr. Baker tells us when the nitrogen trichloride hits the flour it is turned almost completely into hydrochloric acid and ammonia. It seems in both the Industrial Appliance Company's and in the chlorine process, chlorine is the active agent. In that case, I may be very dull, but I can't see why it is so much more chlorine is required in the Industrial Appliance method than in the Agene process.

DR. BAKER: Chlorine is not the active agent in the Agene process. The active agent is nitrogen trichlorid, which is as much different from chlorine as sodium chloride is different from chlorine. It, however, has a property which is unique, in that as a single molecule it possesses six oxidizing units. I don't know of very many oxidizing compounds that have much more capacity than that little compound has, and those six oxidizing units are used in decolorizing the color in the oil. When they are through their work, the final products are hydrochloric acid and ammonia. If you will follow the equations through, you will see that is a fact. It takes six atoms of chlorine to make one molecule of nitrogen trichlorid.

QUESTION: Mr. Chairman, I would like to ask the various bleaching agent men here how many parts per million of active bleaching agents are left in their finished products under ordinary conditions, we will say a hard winter patent flour, all bleached to the same degree, I want to know how much is retained. We are assuming it is all retained. How many parts per million in the finished product? The question has been asked in just what way Agene agrees with natural aging. Now natural aging, in my opinion, is the only real process for aging flour. If it could be obtained by natural agencies, I would prefer that nitrosyl chlorid. Agene and Alsop and that other chlorine man out here—I saw him here a minute ago—Mr. Williams; we haven't heard from him, by the way, yet.

MR. WILLIAMS: I am not interested right now.

QUESTION (continuing): The natural agencies, by natural agencies there isn't but a very small quantity of oxygen retained by the natural oils of flour. I guess all chlorine methods in the oils

of the flour is in the oil in the form of carotin. There is a certain amount of chlorine or oxygen retained by the oil as an addition compound. In my opinion, the Alsop process leaves the smallest quantity of active agent in the flour of any process there is known to man today. It corresponds more closely to the natural agencies than any other process. I would like a statement from those various bleaching agencies just what quantity is left in the flour.

DR. DUNLAP: Well, I can answer that in a very few words. Every bit of chlorine that goes into the flour stays there.

QUESTION: Yes; how many parts per million?

DR. DUNLAP: I tried to cover these points in the course of my little paper. It all depends upon the type of flour being treated.

QUESTION: I spoke specifically upon a type of hard winter wheat, patent flour.

DR. DUNLAP: That all depends upon the type of hard winter wheat patent flour you are talking about.

QUESTION: Kanred wheat.

DR. DUNLAP: I can't tell you.

QUESTION: I would like to ask what quantity of chlorine you advocate using on high patent flour, Kansas wheat.

DR. DUNLAP: That all depends upon the character of flour. It will average—I would hazard this guess on good high grade patent flour, it will average somewhere about 150 parts per million, but I don't care to be bound by that, because it all depends upon the character of flour, but every bit that is used stays there, either in the added form of carotin or hydrochloric acid, but I can't be bound by saying you can take that type of flour, and another type of flour, and still a third type of flour and give them the same degree of treatment because there are so many factors which are involved.

QUESTION: I would like to ask Dr. Dunlap and Dr. Baker whether there is any data regarding treated flour with chlorine after storage, whether it bleaches more in ratio than the flour that is not? Does the flour treated with chlorine after storage bleach more in ratio than the flour that is not?

DR. BAKER: That is impossible because it has not so far to go. It depends upon how much you have bleached it, but naturally the unbleached has much further to go than the bleached flour, and it depends upon how much you have bleached. If you have bleached very far, there is no further to go in improvement; if you have bleached lightly, there is a long ways to go. But in

all cases, you have gone ahead of the naturally aged flour.

QUESTION: Well, one Nebraska miller, who was using chlorine, reported to us that after their flour was treated with chlorine and stored away for a period of time, they said after three weeks they could see a decided improvement in color in ratio to natural flour. In other words, is there some chlorine remaining in the flour in an unattached way that would act on the flour? I would like to know if there is any data on.

DR. DUNLAP: No, so far as I know there is no data. Flour treated with chlorine at once receives the maximum which your chlorine can give it. If the chlorine is not there in sufficient amounts to completely convert your coloring matter into a colorless compound, then there is absolutely no reason why Nature shouldn't continue and do what the chlorine has not done. So it is purely a question of how you have handled your equipment. If your equipment is handled as it should be handled, as a rule you will find very little improvement in color.

QUESTION: Well, this mill reported to us that they had tried this three separate times and the results were conclusive so far as that—

DR. DUNLAP: (interrupting): Well, that may have been, but I can't tell you in the way of actual facts with respect to that particular flour unless I had seen the flour, and I knew how much it should be treated, you see, and how much it had been treated.

QUESTION: Mr. President, may I ask a question of those present? If any one has ever had experience bleaching with Alsop process, getting off enough gas that after a certain length of time the gluten would entirely disintegrate to such an extent it would ruin a loaf of bread? Is that possible with the electric process?

MR. MARMADUKE: It is not.

QUESTION: Now, gentlemen, "them's my sentiments" exactly. I know that the Alsop process has a tendency to make a blue color and a great many chemists have argued with me that it will not produce the same results in patent flour. Now I don't agree with them. I can't see the difference, and I have tried it on two or three crops. Maybe I didn't have the right kind of wheat, but as I spoke to these gentlemen, with the chlorine process I have got to watch around my mill, any mill I happen to go to, all the time to keep this flour from coming back to the mill in carload lots over-bleached.

DR. DUNLAP: I can say to you, with respect to the point you have brought out, this: your having the trouble with respect to chlorine treatment indicates what you need is to have your equip-

ment in proper shape so you will not have that difficulty. At the present time the control machinery of the Industrial Appliance Company gives absolutely no trouble whatsoever. Then your equipment is not in shape. That is all I can say, if you are having trouble with over-treating. It is a question of the equipment not being in proper shape.

QUESTION: Well, Mr. Chairman—

DR. DUNLAP: (continuing): And that is a thing that is very readily put in shape.

QUESTION (continuing): I must grant that your contention is no doubt correct, but on the other hand I must also say, and I believe I can count on the testimony of fully seventy-five per cent. of the chemists, that overbleached flour from chlorine does come back to the mill to such an extent that a great many of us chemists have gotten so that if we bleach at all, we bleach an awful little and certainly not as much as any one of these bleaching processes recommends. Now I was told originally that you could run on a crop of wheat, when we put this in—I won't mention dates (names?), but I do that personally—say with up to two ounces of chlorine. Well, I know from my experience, the last crop an ounce and a quarter on ordinary flour would absolutely kill it.

DR. DUNLAP: What do you use two ounces for then?

QUESTION (continuing): And I have got to go out each and every crop and by the experience of paying for cars of flour find out just how much I can use. Of course we have a control from the representatives of these bleaching machines. What we as chemists want to get away from is this cost to the manager, because every time we have to pay for a car of flour he comes down and jumps onto us, and he should. So all we want is help: I spoke to some gentleman this morning and said we wanted a fool-proof machine. He said, "It don't exist." I grant him it doesn't exist possibly, but we do want a machine that a man of even, let us say, mediocre intelligence, can run.

VOICE: He ought to know enough process. Don't need any machine.

MR. MARMADUKE: I will answer that. The gentleman said with the Alsop process you sometimes get flour blue?

VOICE: We do.

MR. MARMADUKE: Well, you are over-treating it.

VOICE: Well, it has no other harm except that. It gives no other bad effect.

MR. MARMADUKE: No, wouldn't have any bad effect on the flour.

VOICE: You can bleach to such an extent you get all the blue properties without that blue discoloration?

MR. MARMADUKE: Yes.

VOICE: Well, I have something yet to learn on the electrical side. I haven't got your machine at the present time. I will state candidly I would like to have it.

QUESTION: Mr. President, I am not boosting any of these firms, but I agree with Dr. Dunlap, that this man's experience with chlorine in because his equipment is not in shape, because we have had considerable experience with it, and I would say that the Industrial Appliance control is an near "fool-proof" as any on the market. We have absolutely no trouble, except that the agitator once in a while, which is probably due to our own fault, affect just a barrel or two. I would say it is because his equipment is not in the proper shape.

QUESTION: I am pretty much behind the times, but I would like to ask if there is any quick method known for detecting Agene chlorine or Noradel bleach in flour.

DR. MORRISON: I would like to say something in relation to that. I think it has been pretty well demonstrated it is an extremely difficult thing to eliminate sources of error in the detection of artificially treated flour and the chemist who goes into court is rather up against it in proving that he has excluded possible sources of contamination. It is an exceedingly difficult thing to do. I have had a little experience in that and I think that others who have had anything to do with it will bear me out in that statement. There are several methods which may be used, but the chemist who applies them must be one who is thoroughly conscious of the fact that he must preserve the integrity of his results by excluding all possible sources of contamination. There is another thing I would like to bring up. I do not like to orate, but there is one point Dr. Baker made which I think possibly requires—I wont say explanation; that is hardly the thing to say to Dr. Baker—but deserves some comment. Dr. Baker said purity is always associated with whiteness. Now we have heard in the Bible, if any of you ever read in the Bible, of "whited sepulchres," and we also know we can take distilled water and inoculate it with typhus the results will probably wind us up at the undertakers. This may have been a chemical compound which is perfectly white. We might say, take such a white thing as one of the acetic ethers which are among the whitest things I know of, and it is

probably to have there present other compounds, which are also white, but still the general quality present is white, and I think it is not proper, or rather I think in a proper mind the association of whiteness with purity often leads to unpleasant reflections and I think it would be a very good thing to try to point out that whiteness, either of white bread or white flour, necessarily doesn't mean that you have a superior article.

DR. BAKER: I will explain my attitude. I was referring to the opinion of the public, the general opinion that is always dominantly present in the human mind. And it is present. We have white sugar and it is bleached so that it will be whitened and every white article of commercial manufacture, with few exceptions, is made as white as possible. It is an indication of purity even in chemicals.

VOICE: You have distilled water inoculated with—.

DR. BAKER: That isn't white. That is getting away from the absence of color. Unless you get a milky solution.

Whiteness isn't purity, but the point I wanted to make, Mr. Morrison, and I believe it is a very vital point, is the public believes whiteness is purity. I don't say it is purity but they believe it.

MR. MORRISON: The public is misled then.

DR. BAKER (continuing): The public believes it, and I believe they always will believe whiteness is purity.

MR. MORRISON: We sent out a questionnaire to the bakers and they did not agree the whitest bread was always the most popular with the trade.

QUESTION: I would like to ask if either of these two gentlemen would recommend flour be bleached to the extent, with chlorine or Agene, it would not improve in color upon aging?

DR. DUNLAP: I can answer that by saying this, the main objective point in treating flour with choline is not the removal of color. The main objective point is to treat the flour so that you control the intensity of acidity to the point that when you come to the baking, which is the last answer, in the baking of that flour you are going to get the best oven product that that flour can possibly produce, and there, according to the character of the flour, you will have to treat with chlorine in order to develop that tendency of consuming.

QUESTION: I should think the consumption of it would be the latest analysis rather than the baking.

DR. DUNLAP: Well, consumption depends upon the baking, does it not?

QUESTION: Mr. President, I would like to ask Dr. Baker whether the Greiss reagent produces any reaction with Agene treated flour?

DR. BAKER: As I said in my remarks, a minute trace of nitrates developed upon applying nitrogen chloride. In applying the nitrite agent to the flour there is a barely detectable difference between the bleached and unbleached. There is a barely detectable trace. It would be about equivalent to what you get in natural storage in a few weeks, when you get that same development of a faint color.

QUESTION: Mr. President, I would like to ask a question. I won't ask it of you two gentlemen (Dr. Baker and Dr. Dunlap), because they probably wouldn't be interested in giving me an answer. But I would like to know if a solution of potassium iodide will give you any indication as to whether a flour is over-bleached or not with chlorine gas before you bake it? Now my experience has been sometimes this over-bleaching has only been carried to a very slight extent and stands up pretty well under a couple of punches, and breaks down before it goes into the oven, and I was told by a chemist that a solution of potassium iodine would give a distinct coloration if this bleaching has been carried too far, and if it had not and was in proper condition, it would not. I would like to know if anyone has had this experience?

DR. DUNLAP: I have had occasion, in my experimental work, to treat various unbleached flours with varying degrees of chlorine up to the point where the acidity was so largely developed that it was absolutely impossible to wash the particles of gluten from it, and I never yet have been able to punish a flour, to a point, with chlorine, where there is any free chlorine remains, and you must depend upon the presence of free chlorine if you are going to get reaction with potassium iodine. It is absolutely contrary to my experience, any experience I have ever had, and I have probably had as much experience in an experimental way as any of you have had in chlorine treatment of flour.

MR. RAINES: I might say a word along that line. We did use a ten per cent. solution of potassium iodine and when applied to flour bleached with chlorine, even if it only gave a slightly brown tint to that flour, we were unable to get any kind of a loaf or bread out of it at all. If it was treated so far that it gave a distinct blue reaction, really a low black, that the bread was entirely disintegrated when it came from the mixer.

VOICE: May I ask, Mr. Rainey, if that might not be due to a mere iodine test for starch?

MR. RAINHEY: I don't think so, because on a sample of flour known not to be over-bleached, no color whatever was present, not even a slightly brown color.

MR. HESS: When we try that test, we always slick up the flour that we know is not over-bleached right with the flour that is over-bleached, and the one that is not over-bleached will not show discoloration at all.

DR. BAKER: Mr. President, I have experimented on that reaction. You can't extract or take out that yellow color, because you have so much starch and other material present. The color you get is due to the chlorine having formed addition compounds and when the iodide is added, there is an exchange and you get iodide substitute compounds in the place of chloride, which compounds are colored. It is not free iodine nor starch iodine but an iodine exchange compound, which gives that reaction.

At this point, the President declared the session adjourned until Thursday morning, June 8th, at nine o'clock.

SESSION CLOSED.

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### CRUDE FIBER DETERMINATIONS

S. J. LAWELLIN.

Since the Association of Official Agricultural Chemists have gone on record and adopted the double filtration method for crude fiber determinations as their official method a little comparison of methods has appeared to be timely. The American Association of Cereal Chemists in its desire for a rapid, easily adapted, accurate method for crude fiber has chosen the single filtration method with a few modifications. Also a few investigators have stated that the removal of fats is of no especial advantage in crude fiber work while some advocate the removal of fats but claim that they can be removed as well after digestion as before. Our aim in this work was to make a comparison of the single and double filtration methods both with and without the removal of the fats and also a comparison of the single filtration method with and without the modifications advocated by the A. A. C. C.

In the Journal of the A. O. A. C., Vol. IV, (1920), pages 39-41, Francis states that collaborative results reported show that the single filtration method gives high results and does not check the

Official Method of the A. O. A. C. and does not give satisfactory checks between different analysts. As no statements are made as to the acidification of digestate just previous to filtration it is to be assumed that acids were not employed and that digestate was filtered as coming from the digestion. Undoubtedly this would give higher results with the exceptions noted below.

Confirming the views of Henneberg (J. Landwirtschft, 1859, 367), Nolte has said (Z. anal. Chem. Vol. No. 58-392, 1919) that it is immaterial whether the fat is extracted before the substance with the regents or subsequently from the crude fiber. The work of these two authors evidently points out that it is quite necessary to remove the fats before insineration of the sample but that it may be done either before or after digestion with acid and alkali for the preparation of the crude fiber.

Haigh of the University of Missouri has suggested that with feeds high in protein content, such as oil meals, that the sample be digested at 40 degrees Centigrade for thirty minutes in the presence of dilute hydrochloric acid and filtered on asbestos before the sulphuric acid treatment of the Official crude fiber method of the A. O. A. C., no colloidal solutions are then encountered to cause difficulty in the filtration.

It is quite possible that Haigh has used, in a different way, the same modification as adopted in the Official Method of the A. A. C. C. Undoubtedly the digestion with hydrochloric acid just before filtering the crude fiber digestate. By the use of the hydrochloric acid digestion as employed by Haigh the colloids would be prevented effectively from interfering during digestion and might eliminate foaming. However, with ordinary samples encountered in crude fiber work in flour mills the presence of colloids during digestion with acid and alkali do not interfere, so therefore the addition of hydrochloric acid just before filtering would be a more rapid method for elimination of colloids. In fact the treatment with hydrochloric acid may be better just before filtering, as it would then have no effect on the digestion of the crude fiber. When hydrochloric acid is added subsequent to digestion and the sample filtered at once there is little chance for much action by the acid. In as much as we have done no experimenting on samples of high protein content, we would say that Haigh's suggestion may be a good one. However, as the ordinary run of samples in an industrial cereal laboratory do not run excessively high in protein we do not believe the hydrochloric acid should be added until just before filtration. This would save considerable time necessary for the pre-digestion.

In the experiments reported below we have followed very

carefully the methods as outlined and have made all operations standard. In acidifying solutions, just previous to filtering HCl of Sp. Gr. 1.18 has been used and where this reagent has been employed the strength of acid is the only variation from the Official Method of the A. A. C. C. except that in Methods No. 1 and 3 the fats had not been removed. Stronger acid, than is recommended, was used to avoid handling the greater quantity of solutions. As the concentrated acid reacts at once with the sodium hydroxide solution and is also diluted immediately we did not deem this of great significance.

Exact methods used are as follows:

Method No. 1: Two grams of sample are weighed up from finely reduced material and transferred directly, without fat extraction, to one liter Erlenmeyer flasks, sample is boiled exactly thirty minutes with 1.25% sulphuric acid, 200 cc. being used, then without filtering for exactly thirty minutes 200 cc. of 3.52% sodium hydroxide solution. Sample is removed from fire and excess of concentrated HCl, Sp. Gr. 1.18, is added by pouring cautiously down side of flask. Excess is indicated by change of color and breaking up of colloids. Sample is now filtered through alundum crucible of maximum porosity, washed with distilled water until free from acid, washed with alcohol and ether in turn dried, weighed, incinerated and again weighed.

Method No. 2. Method No. 2 is the same as Method No. 1, except that sample is first extracted for sixteen hours with ether in Soxhlet extraction apparatus. Sample is now handled same as under Method No. 1.

Method No. 3. Method No. 3 is practically the same as Method No. 1, only when removing from fire after complete digestion, the sample is not acidified but is filtered immediately through alundum crucible. Sample is thoroughly freed from alkali by distilled water before washing with alcohol and ether.

Method No. 4 Sample is weighed and transferred directly to crude fiber flasks without extraction with ether, boiled exactly thirty minutes with 200 cc. 1.25% sulphuric acid. Sample is now filtered through pure crash linen filter. As much fiber as possible is retained flash and is well rinsed. Fiber on filter is washed free from acid and then rinsed back into Erlenmeyer flask using as little as possible of 200 cc. 1.25% sodium hydroxide solution. Remainder of alkali solution is used in rinsing down sides of flask. Sample is now boiled thirty minutes and filtered hot through alundum crucible.

Method No. 5. Method No. 5 is the same as Method No. 4 or

double filtration method but sample had been previously extracted for sixteen hours with ether in Soxhlet apparatus. Other manipulations remain the same.

These methods were tried out on fourteen samples so chosen as to cover nearly all crude fiber analyses as met with in an industrial cereal laboratory. In the preparation of sample they were ground very fine and all made to pass 100 mesh sieve with the exception of samples containing oats and barley where it was found impossible to reduce the hulls to such a fine state with the apparatus at hand. Previous work by various authors has pretty well shown that the fineness of material makes no appreciable difference in crude fiber results when digestion is thorough. However, for comparison, it was desired, where possible, to have all samples at the same fineness. All samples had been air dried for about two weeks so no moisture determinations were made. The results as obtained are tabulated below.

TABLE OF RESULTS OBTAINED.

Duplicates—	Method 1		Method 2		Method 3		Method 4		Method 5	
	No. 1	No. 2								
Bran .....	11.76	11.50	10.43	10.62	11.80	11.85	10.90	11.00	10.41	10.50
Shorts .....	9.35	9.25	9.40	9.30	10.47	10.42	9.20	9.28	9.35	9.37
Midds .....	6.60	6.03	6.15	5.90	6.23	6.25	6.00	6.18	6.10	6.03
Red Dog.....	4.38	3.94	3.47	3.50	3.39	3.60	3.49	3.82	3.48	3.51
Yellow Hominy	5.65	5.71	4.15	4.07	4.26	4.44	4.38	4.60	4.06	4.09
White Hominy....	3.80	3.74	2.92	2.90	4.00	3.82	3.37	3.70	2.98	3.00
Yellow Fine Feed Meal.....	3.07	3.15	2.33	2.35	3.30	3.80	3.20	3.61	2.37	2.40
White Fine Feed Meal.....	3.62	3.61	2.58	2.56	3.80	4.00	3.27	3.01	2.60	2.55
Yellow Cracked Corn .....	2.40	2.42	1.80	1.83	2.35	2.86	2.27	2.00	1.84	1.82
No. 2 Feed—										
Oats and Corn...	4.06	4.08	3.38	3.30	3.97	4.16	4.02	4.27	3.28	3.25
Rye Midds.....	6.36	6.32	6.35	6.37	6.40	6.50	6.80	6.92	6.40	6.43
Rye Red Dog.....	4.48	4.52	4.42	4.60	4.50	4.85	4.73	4.86	4.50	4.55
Ground Pure										
Oats .....	16.30	16.25	15.70	15.60	16.20	16.40	16.09	16.27	15.50	15.52
Ground Pure										
Barley .....	6.27	6.29	6.02	6.00	6.42	6.67	7.16	7.04	6.04	6.05

A comparison of the analyses will show that by careful work we, at least, have succeeded in securing decidedly satisfactory results and checks between Method No. 2, the Official Method of the A. A. A. C., and Method No. 5, the Official Method of the A. O. A. C. The time saved by use of Methods No. 2 was considerable when the full battery of six samples were run. With Methods No. 2 and No. 5 the finished crude fiber was equally as clear and free from impurities in each case and no color differences were noted. The results of Methods No. 1, No. 3 and No. 4 were, in nearly all cases, much higher. This was as expected, as the fats were

not removed either before or after digestion of sample for crude fiber. These methods need not be considered of any value, but a few observations may not be out of place.

In Method No. 3, the single filtration method where no HCl was added previous to filtration, the filters quickly clogged and filtering became very difficult. This was due to gels or colloids present and they formed an almost impervious mass in crucibles. These samples took considerable time to filter and wash, and apparently samples could not be freed entirely from gels by washing. Washing with ethyl alcohol and ether removed some gel, but not all. Repeated washing with these reagents did not appear to remove gels beyond a certain point. Gels seemed to be of three natures, water soluble, acid soluble, and alcohol and ether soluble. Remaining residue was of dark color and jelly like to some extent. In this method the worst trouble was encountered with the wheaten bran. One exception was noted, and that was in samples containing oats. In samples consisting of oats alone no gels were formed by any method, but will be formed in the single filtration method if strong sulphuric acid is used to acidify digestate subsequent to digestion and just prior to filtration. The sulphuric acid, of the strength used, employed in first boiling of oat samples apparently was too weak to cause formation of gels to any extent.

In the case of corn samples with Method No. 3, there was a very slight amount of gel formed, but this, however, did not hinder filtration with the single filtration method, where fats were not removed and digestate was not acidified after digestion. However, when samples were being washed with ether, after thorough washing with distilled water and alcohol, a considerable amount of gel formed, which could only be removed by washing with hydrochloric acid.

Method No. 4 gave a fine looking fiber, apparently free from gels and other harmful bodies, but results were generally higher than with either Method No. 2 or Method No. 5. Samples were easy to filter and wash. In a comparison of Methods No. 3 and No. 4, the time lost in the double filtration Method No. 4 was regained when final filtration and washings were made. In the second boiling of the double filtration Method No. 4, where fats were not removed, considerable trouble was encountered by foaming of the digestate. This did not occur so freely in any of the single filtration methods.

**Conclusions.** The results obtained in this work show that the single filtration method as the Approved Method of the Amer-

ican Association of Cereal Chemists, is fully as reliable as the double filtration Official Method of the Association of Cereal Agricultural Chemists. Under standard conditions and with careful work, satisfactory checks can be obtained between the two methods.

The great saving in time and operations secured by the A. A. C. C. method makes it the more efficient and better method for Industrial Cereal Laboratories. The single filtration method is impractical unless, except in cases of oats, when digestate is not acidified just previous to filtering. Hydrochloric acid is the best acid, so far recommended, to use for acidification of digestive. thought sulphuric acid may be used except in the case of oats.

The removal of ether solubles is necessary to obtain correct results, and it is recommended that this be done prior to treatment of sample by acid and alkali, as in digestion of sample for crude fiber determinations.

*EAGLE ROLLER MILLS,  
New Ulm, Minn.*

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#### ASH ON FLOUR.

J. R. HESS.

Much has been written about the ash on flour and mill products in the past, but very little has been said of the manipulation and source of error in the determination. In running an ash, whether the amount used for determination is one gram or twenty, there are sources of error that many chemists overlook.

#### Proper Sampling and Keeping of Samples.

Taking a sample is, at its best, very haphazard but, the way it is actually taken in most cases, is enough to give a chemist a chill when he thinks of the accuracy that is demanded of him on this determination. It is indeed strange that we check as close as we do. Samples are taken with a scoop of more or less freedom from dust, each particle of which may throw you off the famous .01 or .02 per cent, or, as I was told once, 1 or 2 per cent. The sample for analysis should be taken with the greatest care, if close checks are demanded and given. I have small glass salve jars, that are easily cleaned and wiped out, for the analysis, and as an additional precaution I always scrape the top flour off. These are so small that it's foolish to use a dirty scoop to fill them,

and as a result we get a pretty fair sample for analysis. I have had samples sent to me in buckets that had chaff and dirt all over the top, and I couldn't tell how much had been stirred in. Then I had glass jars that sealed and found pieces of rubber, dust, etc., on top of flour, so finally the little salve jars were adopted, and we still use the sealed jars for the baking test. The little cans have been so gratifying that I like recommending them to everyone who has been annoyed by not being able to obtain checks. A little dirt can surely throw you off. See results in Table 1. An ash test should never be run without a moisture test, also, and in reporting results I always give ash and moisture.

TABLE I.  
Effect of improper cleaning and ignition.

Crucible No.	Improper Cleaning	Clean
1	.33	.36
2	.605	.365
3	.72	.37
4	.395	.37

#### The Kind of Crucibles Used.

I have tried plantinum crucibles, but don't remember any special advantage in using them. Moreover, they are out of the question for most of us on account of price. Silica, both opaque and clear, are the best crucibles to use. The material burns faster and is less liable to fuse. In fact, I have never had an ash fuse in my silica crucible, although it probably could be done, but at ordinary temperatutes used for the ash determination no fusion is experienced. The material will not fuse so readily as in the porcelein, and you get a white ash much easier. I have been trying out my silica crucibles for some months now, and believe I can recommend their use in preference to anything else. I have tried other metal crucibles, but found them unsatisfactory.

#### Properly Cleaned Crucibles.

If previous ash has been burned properly, the ash is fluffy and can be brushed out and crucible used again. If crucibles are kept in the dessicator and weighed up just before flour is added, no noticeable error can be detected if used in this manner. but if the ash sticks or fuses in the bottom and can not easily be removed with a brush, then they must be treated with acid, preferably HCl (diluted), washed out and rinsed with distilled water, dried and ignited at high temperature and then placed in the desiccatior; when COOL to room temperature weigh and proceed with the determination. I have known chemists to wash

rapidly with tap water after treatment with acid and then dried and heated slightly, and used. Table 1 shows some of the errors that have been noticed by such proceedings. This plainly shows the importance of proper cleaning of crucibles.

#### Weighing of Sample.

The weighing of samples, of course, should be done with accuracy whatever amount is used. I have tried all the way from 1 gram to 5 grams, and even with 1 gram have obtained duplicates in order. These are suggested by an incident which occurred within .01 per cent as ash, also on 2, 3 and 5 gram samples. So as to the amount of sample, it is a matter of choice, or time allowed for the determination. The procedure must be different for different amounts. The 1 gram sample will get gray much sooner than 5 grams, so I have adopted 2 grams so as to cut my error as much as possible and still get the ash burned within the time limits allowed. Last year (1921) we used 5 grams with as rapid results as we get now on this year's crop with 2 grams. After one has obtained the weight of the crucible, which must be weighed to within the accuracy of balance. The weighing of the flour is a simple proceedings and should be rushed as much as possible, as we know flour gains or loses moisture very rapidly after a short time of exposure to air. The weighing of the flour causes less error than any other part of the determination. For a 1, 2, 3, or 5 gram sample of flour an error in weighing of .001 gram makes no weighable error in the final ash. (See Table II). An error this large is practically impossible if any care is taken at all. On my balance one full scale reading is equal to .0003, therefore, if I allow my pointer to be either three scale readings greater to the left or right I would not get any difference in final results. However, I always weigh to less than one scale reading, so the error due to weighing of sample should be "infinity." (See Table III). These were tests made in our laboratory.

After weighing sample the next step is the burning.

TABLE II  
Error due to weighing of sample (Calc.)

	Patent			
Exact	1gm.	2gm.	3gm.	3gm.
	.37	.37	.37	.37
+ .001	.3696	.3698	.3698	.3699
— .001	.3703	.3701	.3701	.3700
	Clear			
Exact		.600		
+ .001		.5993		
— .001		.6006		

TABLE III.

Actual trial on two gram sample for error due to weighing of sample.

	Four Scale Readings Heavy	Exactly 2 Grams
1st	.37	.37
2nd	.365	.37
3rd	.600	.595

#### Proper Burning of Material.

Regardless of whether you have a pyrometer or not, there is a certain definite way of burning an ash, namely: Start at low temperature and after ash is burned to a grayish color, raise the temperature so as not to fuse but high enough to give a white, fluffy ash. The only way to do this is by trial, and then play safe for the weather conditions seem to affect the burning, at least, as done at present by most of us in the electric muffle. This would apply whether you have a pyrometer or not. I have found two small variations of procedure in running ashes that help. Place crucibles on small fire clay tripods (taken off the ordinary inverted gas mantle) in the furnace. The crucibles fit these holders very nicely, and are thus held from direct contact with the bottom of furnace and have less tendency to fuse. Another kink which has been tried out in our laboratory for several months with good results, is: After ash has become gray or spotted gray and black, remove them from the furnace and place in dessicator for about five minutes, then add about one-half cc. of distilled water and break the ash and spread over the bottom of crucible by tapping on the outside or by swirling the water. Dry and replace in the furnace, which has been closed in the meantime, to get added heat. Also raise the heat by use of the rheostat. Leave in the furnace about an hour at this high heat. The ash is burned white and does not fuse. They will fuse, of course, but they will stand considerable more heat than the fluffy ash. In this way I can get an ash burned out in about three hours. In fact, this year I had to burn my ashes at a very low heat for about twenty-four hours to get a white fluffy ash. The ash fused easier than usual, and as a consequence had to use a lower heat than formerly.

We now come to the point of greatest error in the determination of ash, that is the weighing up of ash after burning.

#### Reweighting.

The ash must be COOLED TO ROOM TEMPERATURE before removal from the dessicator. The effect of the short cooling time usually used and not getting the ash cooled to room temperature before placing on the balance is our greatest error.

See Table V. This shows some variations on a patent and straight grade run on the same day. The time of cooling varies from day to day; some days thirty minutes is sufficient, other days three hours is not enough. I have a thermometer in my dessicator with mercury bulb on a level with the crucibles. In leaving ashes in dessicator over night after cooling to room temperature if they gain in weight your dessicator is not efficient. The dessicator must be efficient for use. This can be tested easily in this manner. I found all our dessicators were inefficient. We were using  $\text{Ca Cl}_2$  as a dessicant. Calcium Chloride may be efficient at first, but sulphuric acid is the only one tried that gave results. I have weighed up an ash, then replaced in my dessicator and left over Sunday, and on reweighing on Monday morning found absolutely no variation. So be sure to have dessicators efficient and cool the ashes to room temperature, and much of your trouble with ashes will be over. As to time consumed in weighing, contrary to the popular notion among chemists, the ash of flour when properly burned takes up moisture very slowly. I have timed myself and ordinarily I can weigh up an ash in  $1\frac{1}{2}$  minutes, or less. After about five minutes the ash gains slightly, but not over .0001 in ten minutes the gain amounts to just .0001. I have tried this not once, but many times.

TABLE IV.

Error due to weighing at temperature higher than room temperature.  
Room Temp.  $105^\circ$ .

Time	15 min.	30 min.	1 hr.	2 hr.	Over Night
1st Trial	.34	.345	.35	.37	.37
2nd Trial	.309	.398	.405	.425	.43

## SUMMARY.

1. Take care in taking of sample.
2. Have crucibles clean.
3. Burn slowly.
4. Cool in efficient dessicator to ROOM TEMPEARATURE if you have to leave over night.

THE WILLIAM KELLY MILLING CO.,

Hutchinson, Kansas.

**CHEMICAL TEST FOR HEAT DAMAGE IN WHEAT.**

(This test was described by the author in March 10, 1922 issue of the *Grain Dealers Journal*.—The Editor).

The Test for gluten, is also a test for heat damage in wheat. The test consists in making a tiny "dough" out of one to two milligrams of flour on a glass slide by the addition of a drop of eosin solution (0.2 grams water soluble eosin in 1000 cc distilled water) and gently rubbing or mixing the flour and liquid with a second piece of glass. Note the formation of carmine colored "gluten roots" due to absorption of the eosin by the gluten if the kernel is sound, and the red specked appearance of the "mealy" dough (devoid of gluten roots) when made from a heat damaged kernel.

Heat, when it causes "heat damage," destroys the adhesive or dough making quality of the gluten. It is necessary to remove the bran and germ before grinding each kernel in a mortar. If both ends of the kernel are cut off it is comparatively easy to peel the bran from the large portion of the kernel.

W. L. FRANK,  
Sherman, Texas.

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**NOTICE.**

The Sample and Referee Committee have received reports covering 95 per cent of the samples forwarded the members of the Association.

Tabulation of results is practically completed, and collaborating analysts will receive mimeographed copies in a short time.

The committee urges more promptness in reporting results. Fully 50 per cent of the reporting members on the first sample reported within seventy-two hours. The remaining reports are

Sample No. 2 will be sent out on the 20th of October.

RALPH S. HERMAN, Chairman.

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